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QUALITATIVE ANALYSIS



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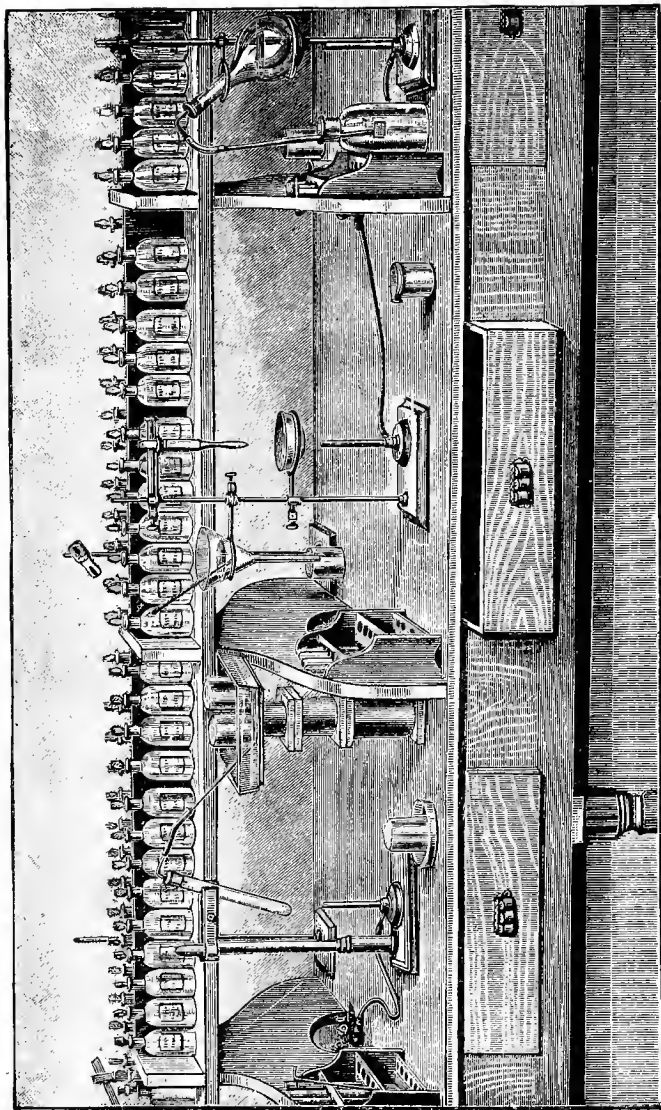
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STUDENTS' TABLE FOR ANALYTICAL WORK.

[See page 74 for description.]

A PRACTICAL COURSE
IN
QUALITATIVE ANALYSIS,
FOR USE IN
HIGH SCHOOLS AND COLLEGES.

By JAMES W. SIMMONS,
SUPERINTENDENT OF CITY SCHOOLS, DOWAGIAC, MICHIGAN.

WITH ADDITIONS FOR STUDENTS' WORK,

By LA ROY F. GRIFFIN,
PROFESSOR OF PHYSICAL SCIENCE, LAKE FOREST UNIVERSITY.

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PREFACE.

THIS work is based upon the author's own experience in the class-room, and is not designed to take the place of our ordinary text-books on Chemistry, but to supplement them.

But few of our Chemistries in common use make any pretence toward practical Qualitative Analysis. To meet this want, the author has collected notes from numerous sources, especially from class-room observations, and now presents the best of them in this form.

Great care has been taken to classify the subject-matter thoroughly, and to present the simplest and most practicable tests to the student, and to adapt the work to the wants of those schools that are supplied but moderately with apparatus; at the same time, however, sufficient material is given to instruct the student thoroughly in laboratory practice, in our best institutions.

Care has been taken in the naming of precipitates, in forming reagent solutions, and in the description of apparatus. Especial attention is called to the Solubility Table, and to the Group Reagents and Preliminary Tests for both metals and acids; also, to the convenient form of arranging the Tests, and the manner of forming Reagents.

The additions for students' use, by Professor La Roy F. Griffin, are those portions in smaller type having the letter (*G*) affixed. The author is greatly indebted to Professor Griffin, and others, for valuable suggestions.

The following authorities have been employed in comparing results: Roscoe and Schorlemmer, Bloxam, Attfield, Kedzie, Shepard, Avery, Stoddard, Rolfe and Gillett, Rains, Barker, The Boston Journal of Chemistry, and various others.

. W. S.

DOWAGIAC, MICH., August 10, 1887.

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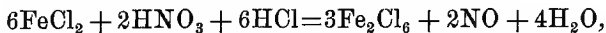
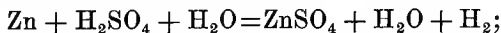
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INTRODUCTION.

IN many schools Chemistry is studied from a purely theoretical standpoint.

Much is said and studied about *what may be done*; and yet, in only a few schools, do the pupils go to work and do the things described. One great hinderance to qualitative work in Chemistry has been a lack of subject-matter in a proper shape for the pupil's use. This book is intended to meet that very want. The tests are such as can be given with clearness and attractiveness before a class, by either teacher or pupil, and they are of equal value when every pupil works entirely by himself.

Each pupil should be supplied with a copy of the work, and then have assigned to him definite lessons for study. It will be a valuable exercise to have the pupil bring to the class a statement of the test reactions written in the form of equations with the equations properly balanced. For example,



show the balancing of chemical compounds after reaction, and tests are balanced in the same way.

It is a good plan to use this work in connection with ordinary descriptive texts. First, study carefully the *occurrence, preparation, properties, and uses* of any metal or

acid; then, follow that by giving a thorough line of tests for that substance.

The author of this work has prepared a blank book of "One Hundred and Ten Experiments in Chemistry," to accompany this book or any work on general chemistry or chemical analysis. The main feature of the book is that an experiment is indicated, and the pupil or teacher is to go forward and make the same, the class carefully noting in the blank space, the *manipulations* of the experiment, their *observations* and the *conclusions* which they may draw from the same. This work of experimentation, conducted systematically, and upon scientific principles, is a never-failing source of interest and profit to all engaged in it. The blank book referred to can be obtained from the publishers of this work.

The natural sciences should be taught mainly through illustration and practice in the Laboratory. Chemistry can not be profitably pursued in any other way than by illustration through experiments, and as large a part of this as the circumstances permit should be performed by the student-himself.

Do not wait until you have a well stocked Laboratory before you begin to experiment, but get a few things and go to work, and your laboratory material will increase in direct ratio to your interest. The materials are comparatively inexpensive and few in number.

A PRACTICAL COURSE IN QUALITATIVE ANALYSIS.

CHAPTER I.

PREPARATORY DEFINITIONS AND DIRECTIONS.

Qualitative Analysis is the determination of the elements in a compound.

Quantitative Analysis is the determination of the quantity of the individual elements in a compound.

An Acid is a compound of hydrogen, possessing certain properties, chief of which are a sour taste, turning vegetable colors red, and allowing a metal to take the place of a part or the whole of its hydrogen. (Insoluble acids have no taste.)

All acids contain hydrogen, but not all bodies containing hydrogen, are acids.

Nearly all acids contain oxygen, and are called ox-acids. In naming ox-acids of a single element, *ic* denotes the stronger and *ous* the weaker; as, sulphuric acid, H_2SO_4 ; sulphurous acid, H_2SO_3 . When there are three acids, the intermediate in strength is usually named by some combination of the names of the elements themselves. When there are four, the weakest has the prefix *hypo* and the

strongest *per.* When there are more than four, some of them are given special names.

ILLUSTRATIONS: HClO , Hypochlorous Acid.

HClO_2 , Chlorous Acid.

HClO_3 , Chloric Acid.

HClO_4 , Perchloric Acid.

A Base is usually a compound of some metal, and it always has the property of neutralizing acids. It does this by replacing the hydrogen of the acid with the metal of the base.

Oxygen and hydrogen unite with some elements to form bases and with others to form acids.

With few exceptions, the compounds of hydrogen and oxygen with the metals are bases.

The soluble and stronger bases are alkalies.

Usually, the union of oxygen and hydrogen with the metallic elements forms bases, and the union with the non-metallic elements forms acids.

ILLUSTRATIONS: KHO , Potassium Hydroxide (base).

NaHO , Sodium Hydroxide (base).

$(\text{H}_4\text{N})\text{HO}$, Ammonium Hydroxide (base).

H_2SO_4 , Sulphuric Acid.

HNO_3 , Nitric Acid.

H_3PO_4 , Phosphoric Acid.

A Salt is the compound made by the union of a base and an acid. The hydrogen of the acid is replaced by the metal of the base.

Sometimes the acid is not wholly neutralized when united with a base, and the compound still retains acid properties.

An Acid Salt is one in which only a part of the hydrogen has been replaced.

A Normal Salt is one in which all of the hydrogen has been replaced.

Sometimes the base is not wholly neutralized when united with an acid and the compound retains basic properties.

A Basic Salt is one in which only a part of the metal has been replaced.

Sometimes a part of the hydrogen of an acid is replaced by one metal and a part by another.

A Double Salt is one in which the hydrogen has been replaced by more than one metal.

$K_2 + 2H_2SO_4 = 2KHSO_4 + H_2$, the salt being acid.

$Zn + H_2SO_4 = ZnSO_4 + H_2$, the salt being normal.

$ZnCl_2 + Zn(HO)_2 = 2ZnClHO$, the salt being basic.

$H_2CO_3 + KHO + NaHO = KNaCO_3 + 2H_2O$, the salt being double.

The salt of an *ic* acid takes the ending *ate*; of an *ous* acid, the ending *ite*.

ILLUSTRATIONS: $Ba + H_2SO_4 = BaSO_4 + H_2$, sulphuric acid giving barium sulphate.

$Ba + H_2SO_3 = BaSO_3 + H_2$, sulphurous acid giving barium sulphite.

Nitrates are formed from nitric acid.

Nitrites are formed from nitrous acid.

ILLUSTRATIONS: $2Ag + 2HNO_3 = 2AgNO_3 + H_2$, the salt being silver nitrate.

$2Ag + 2HNO_2 = 2AgNO_2 + H_2$, the salt being silver nitrite.

CHAPTER II.

APPARATUS AND REAGENTS.

APPARATUS.

Although very convenient, yet it is not absolutely necessary to have all of the appliances of a modern laboratory, in order to do satisfactory work. We give the essentials for a laboratory in which all the tests named in this volume can be performed, and as many additional experiments for practice as are required to give the pupil a proper knowledge of the subject.

Any teacher can easily modify this list where the pupils are divided into groups for class experimentation.

Where the circumstances will admit, and individual work is impossible, we suggest the dividing of a class into groups of four, for practice in the manipulation of experiments.

1. **Test Tubes.** Get one dozen four-inch test tubes on broad foot; also, one dozen five-inch tubes without the foot. Two eight-inch test tubes can occasionally be used. For individual work, use the test tubes without foot.

2. **Swab.** A swab for cleaning tubes can be made by fastening a piece of sponge to a small stick.

3. **Lamp.** Where the Bunsen burner is not obtainable, the alcohol lamp will do very good service. One can be made by putting a short piece of glass tubing through

the cork of a quinine bottle, and then drawing the wick through the tube. When additional heat is required, a conical sheet-iron chimney, four inches long, can be put over the lamp.

4. Glass Tubing. One pound of assorted, soft-glass tubing, ranging in diameter from one-eighth to three-eighths of an inch, will be amply sufficient.

5. Test Tube Rack. A rack for holding test tubes is cheaply made by nailing together pieces of pine board, and boring holes for the tubes. It should hold twelve. A row of pins upon which to dry the tubes is a convenience. Have the lower board three and a half by twelve inches, and the upper board two by twelve inches, with end pieces three inches long. Bore holes in the upper board, and set the pins in the front edge of the lower board.

6. Water. When not otherwise supplied, water can be furnished from a cask resting upon an elevated bracket, the water being conducted to and a little above the wash-basin, by means of suitable tubing.

7. Wash Bottle. Take any bottle of convenient size, having a large mouth. Fit a cork tightly, using one with two holes through it. Use glass tubing of about one-fourth inch outside diameter. Have one piece about six inches long, slightly bent about two inches from the end. Place the shorter arm through the cork. The other piece should be long enough to reach from the bottom of the jar up through the cork, and project about six inches, bent at a downward angle above the cork, and having the outer end drawn to a fine point. Place distilled water in the bottle, blow through the shorter tube, and a spray will pass

through the other. This is used in washing precipitates, where a gentle stream is wanted.

8. Rubber Tubing. About three feet each of three sizes of rubber tubing, corresponding to the smallest, medium, and largest sizes of glass tubing, will be needed. Rubber tubing should be a trifle smaller than the glass tubing with which it is used.

9. Corks. A few assorted sizes of rubber corks pierced with two holes, also a few common corks, carefully selected as to compactness, will be required in the work.

10. Luting. Paraffine, beeswax, and sealing-wax are the articles commonly used.

11. Gas Duct. A gas chamber or duct to convey from the operator all poisonous or disagreeable gases, such as chlorine, hydrogen-fluoride, phosphuretted-hydrogen, and arseniuretted-hydrogen, will be almost a necessity. It can be a three-inch tin tube expanding into a funnel over the operating table, the opposite end terminating in some hot-air flue.

12. Platinum. One foot of fine platinum wire for flame tests, and a piece of platinum foil one inch square, will be sufficient.

13. Files. A good, sharp, triangular file for glass cutting, and a medium sized "rat-tail" file for boring corks, will be needed.

14. Blow-Pipe. A common form of the instrument known as Black's, is a good one as well as cheap. A fair one for ordinary purposes can be made by drawing a piece of glass tubing to a fine point and bending it at a right

angle about two inches from the point. Those forms used by jewelers are among the best.

15. Blue Glass. Two lights of blue glass, eight by ten inches, set in a wooden frame so as to make a double thickness, will be wanted in making the "flame test." A piece of cobalt blue glass, three by four inches, will sometimes be needed.

16. Glass Working. Glass tubing can be cut by first making a transverse scratch with a file, then placing the tube in both hands, with the mark between the thumbs; a quick pressure will produce an even fracture. Soften the tube in the alcohol flame when it is desirable to change its form or to make the rough edges smooth.

17. Graduates. A lipped metric jar, capacity of one hundred cubic centimetres, graduated to one cubic centimetre, also an English fluid measure, capacity two ounces, will be used in measuring liquids.

18. Pipettes. Three or four will be needed. They can be purchased, or they can be made from pieces of glass tubing, partly drawn to a point in the alcohol flame.

19. Filter Papers. These can be obtained at any drug store. Three and a half or four inch circular filters are most convenient, but the ordinary filter paper in sheets will answer.

20. Litmus Paper. This can be purchased, or can be made by dipping unsized paper into a solution of litmus, prepared by macerating litmus in water. The red can be made by dipping the blue papers into a very weak solution of acetic acid. Keep the papers well secured in a box, or, better, in a glass-stoppered bottle, when not in use.

It can also be made as follows:

Boil litmus with about six times its weight of water, and filter. Divide the filtrate into two parts. Into one, if the color be not already blue, stir a few drops of a solution of sodium hydrate, or just sufficient ammonia to give the solution a decided blue color, and into the other drop hydrochloric acid, cautiously, until the liquid becomes red, taking care to cease dropping in the acid as soon as the solution has become decidedly red. One solution is now blue and the other is red. Strips of unsized paper, as blotting paper, are now dipped into the solutions, and then dried, by hanging on or by threads. Keep the paper well protected from chemical fumes and from the light. In using for tests, the blue paper is reddened by acids and the red paper is blued by alkalies. Sometimes it is better to moisten the paper in distilled water before making the test.

21. Turmeric Paper. Repeatedly digest the powdered root in small quantities of water to remove objectionable coloring matters, and lastly treat it with dilute alcohol, and filter. Into the alcoholic solution thus obtained, dip strips of white unsized paper, as printing paper; dry and keep from the light. The papers are yellow. Alkalies and boracic acid turn the paper brown, but liquids containing boracic acid may be distinguished by the fact that if the paper be afterward dipped into a solution of hydrochloric acid the yellow is not restored, while it would be in case the change of color had been produced by an alkali. Turmeric paper is also used as a sensitive test in other ways.

22. Mucilage for Labels. Macerate five parts of good glue in eighteen parts of water, for one day. Add

nine parts rock-candy and three parts gum-arabic. The mixture can be brushed upon the paper while it is lukewarm. If it is to be kept in damp places, prepare a paste of rye flour and glue, to which linseed oil, varnish, and turpentine have been added in proportion of half an ounce each to the pound.

23. Starch Paste. Macerate a little starch in cold water, after which add boiling water to bring it to the required degree of fluidity. Starch paste containing KI is needed to test for nitrites, and is also used as a test for ozone. It is prepared in the same way, only that one or two crystals of KI must be dissolved in the water. It can be kept for some time in a glass-stoppered bottle.

24. Funnels. Two or three different sizes of glass funnels will be found serviceable.

25. Mortar. A Wedgewood mortar, medium size, will be required.

26. Evaporating Dishes. Of these, get one of two ounces capacity and one of four ounces capacity.

27. Beakers and Test Glasses. Get two glass beakers, capacity one and two ounces; also two conical test glasses, capacity one ounce.

28. Balances. There are several forms of these. It is not necessary to get an expensive instrument, but something which will weigh with approximate accuracy from one grain to two ounces.

29. Ring Stand. A stand of three rings will be needed for supporting evaporating dishes, retorts, etc.

30. Test Tube Holder. For quick work, use a folded strip of paper clasped around the upper part of the

tube. For more permanent work, make a stand with wooden upright, from which extends an arm, either split or with hinged piece, slightly notched toward the end, for holding the tube, and clasped by a rubber band. The arm should be so constructed that it may be moved up or down on the upright.

31. Flasks or Retorts. Procure two or three Florence flasks, assorted sizes. Flasks and beakers should be heated over the sand bath or upon wire gauze. In heating flasks, beakers, or other glass vessels, care should be taken not to apply the heat suddenly at one point, but to distribute it as evenly as possible over the surface of the glass, thus avoiding unequal expansion and breakage. This may be accomplished by moving the vessel about over the flame, or, better, by applying the heat indirectly by means of a sand bath, or by supporting the vessel on a piece of wire gauze and applying the heat to that.

32. Wire Gauze. Use what is called "strainer cloth."

33. Sand Bath. Use an iron dish, saucer-shaped, filled with fine sand. Place it on the ring stand and set the retort on the sand. Apply the flame beneath the dish.

34. Charcoal. Procure some selected pieces of charcoal of fine grain; cross sections of small limbs preferred.

REMARKS.—The taste, means, and facilities of the operator will naturally dictate the kind and amount of apparatus needed. Much of it can be "home made," and thus materially lessen the expense. Cases will be needed in which to keep apparatus and chemicals free

from dust and from careless handling. Get distilled water from the condensing pipe of some engine.

When each pupil works by himself, a sufficient amount of apparatus must be supplied to furnish test tubes, flasks, evaporating dishes, ring stand, funnels, sand bath, and tube stands to each pupil. The other pieces can be used in common.—*G*.

REAGENTS.

A **Reagent** is a substance used in the determination of the elements of an unknown compound.

Some reagents are used in the dry form, but most of them are in the form of solutions.

Use *distilled water, pure chemicals*, and keep the apparatus *scrupulously* clean.

The commercial chemicals often contain many impurities which spoil the accuracy of the test, yet they may be conveniently used when great accuracy is not essential, as they are much less expensive than those which are chemically pure.

In using reagents, *add cautiously, proceed carefully*, and keep your eyes "*wide open*." A single drop often makes a great difference in the results.

In using reagents, great care must be taken that the stoppers of the bottles do not become changed. Do not use the same dropping-tube in two or more reagent solutions. Corks can be paraffined to prevent sticking. If corks stick, take one or two twists about the neck of the bottle with a strong cord. A sawing motion will produce sufficient heat for expansion.

Note.—The most convenient proportions for actual work are given in the following solutions. Usually, it is not essential that these exact proportions be followed. More or less of the solution can be added according to the strength.

When the laboratory is fitted for individual work, the common chemicals should be provided for each student and placed on a stand in front, upon the work table. Of those less commonly required, indicated by a star (*), a single bottle of each can be prepared and kept where they are accessible to all the students.—*G*.

AgNO₃, Silver Nitrate. Dissolve 1 part of the salt in 50 parts of water.

BaCl₂, Barium Chloride. 1 part of the salt to 10 parts of water.

CuSO₄, Copper Sulphate. 1 part of the salt to 8 parts of water.

CaSO₄, Calcium Sulphate. A saturated solution.

FeSO₄, Ferrous Sulphate. 1 part of the salt to 10 parts of water.

***Fe₂Cl₆, Ferric Chloride.** 1 part of the salt to 15 parts of water.

H₂SO₄, Sulphuric Acid. Concentrated, C. P.

(H₄N)₂CO₃, Ammonium Carbonate. 1 part of the salt, 4 parts of water, and 1 part of ammonia solution.

HCl, Hydrochloric Acid. Concentrated, C. P.

Acids are often used in diluted forms.

Dilute the acid with about four times its volume of distilled water.

H₂S, Hydrogen Sulphide. Through a cork in a wide-mouthed bottle, insert a glass tube of one-half inch diameter and about six inches long. Melt the lower end of the tube and nearly seal it, leaving a hole about the size of a knitting needle. Have a rubber delivery tube attached to the upper end of the glass tube, the opposite end terminating in a small glass delivery tube. Have a solu-

tion of H_2SO_4 and H_2O in the jar, and some fragments of FeS in the glass tube. Lower the tube into the acid solution and H_2S will be freely liberated. Raise it when you wish the flow to be stopped. A saturated solution of H_2S in H_2O is often used, but the fresh gas is much better. Remember that the gas should not be breathed.

$(\text{H}_4\text{N})_2\text{S}$, **Ammonium Sulphide**. This solution is generally purchased ready for use. It can be prepared by passing a current of hydrogen sulphide gas into dilute aqua ammonia until the solution will not precipitate magnesium sulphate.

H_3N , **Aqua Ammonia**. The reagent solution usually contains 10 per cent. of gas. The ammonia of the shops, 28 per cent. Dilute the ammonia of the shops with three volumes of water.

* HgCl_2 , **Mercuric Chloride**. 1 part of the salt to 18 parts of water.

$(\text{H}_4\text{N})\text{HS}$, **Ammonium Hydrogen Sulphide**. This can be purchased, or it can be made from $(\text{H}_4\text{N})_2\text{S}$ gently warmed in a test tube with a small quantity of flowers of sulphur. It is sometimes called "yellow ammonium sulphide."

$(\text{H}_4\text{N})_2\text{S}$ allowed to stand exposed to the light, turns to $(\text{H}_4\text{N})\text{HS}$; usually this becomes a sufficient source of the reagent.—*G*.

* H_2SiF_6 , **Hydrofluosilicic Acid**. Prepared by dissolving 3 parts of SiF_4 in 4 parts of water.

$(\text{H}_4\text{N})_2\text{C}_2\text{O}_4$, **Ammonium Oxalate**. 1 part of the crystallized salt in 24 parts of water.

$\text{K}_2\text{Cr}_2\text{O}_7$, **Potassium Bichromate**. 1 part of the salt to 10 parts of water.

* **KI, Potassium Iodide.** 1 part of the salt to 20 parts of water.

* **KHO, Potassium Hydroxide.** 1 part of the dry sticks to 20 parts of water. Filter the solution if not clear.

* **KCy, Potassium Cyanide.** 1 part of the salt to 4 parts of water. Remember the exceedingly poisonous nature of this solution.

K_4FeCy_6 , Potassium Ferro-Cyanide. 1 part of the crystallized salt to 12 parts of water.

* **K_3FeCy_6 , Potassium Ferricyanide.** 1 part of the salt to 12 parts of water. This solution rapidly deteriorates, and had better be prepared freshly from the crystals when required for use.

* **K_2CrO_4 , Potassium Chromate.** 1 part of the salt to 10 parts of water.

K_2CO_3 , Potassium Carbonate. 1 part of the dry salt to 10 parts of water.

$Pb(C_2H_3O_2)_2$, Lead Acetate. 1 part to 10 parts of water.

Na_2PHO_4 , Sodium Phosphate. 1 part of the salt to 10 parts of water.

NaHO, Sodium Hydroxide. 1 part of the dry sticks to 20 parts of water.

Na_2CO_3 , Sodium Carbonate. 1 part to 5 parts of water. Often used in the dry form.

* **$PtCl_4$, Platinum Chloride.** 1 part of the salt to 10 parts of water.

$H_6C_4O_6$, Tartaric Acid. Strong solution in water.

CaCl_2 , Calcium Chloride. 1 part of the salt to 8 parts of water.

Note.—The reagents named are sufficient for the tests given in this book. They should be put up in two-ounce bottles of uniform size, with ground-glass stoppers. A piece of rubber tubing should be pushed over the stoppers of all the bottles which contain strong alkalies, else the reagent will soon act upon the glass and prevent their removal. To use small quantities, employ the dropping-tube or pipette. Have separate pipettes, or *thoroughly* clean one before putting it into a different fluid.

CHAPTER III.

PRELIMINARY EXAMINATIONS.

FLAME TEST.

INDICATIONS OF COLORED FLAMES.

A *clean* platinum wire moistened with HCl and dipped into the powdered substance, and held in the flame, shows colors as follows:

1. Blue, changing to green, indicates Cu, Sb, Pb, As. (Except with Cu, green is nearly wanting.)

Note.— Never dip a platinum wire into a substance suspected to contain As; it dissolves the wire.— *G*.

2. Crimson indicates Sr.

3. Green indicates H_3BO_3 , Boric Acid.

4. Violet indicates K. This is shown through blue glass, which cuts off the sodium colors.

5. Yellowish-green indicates Ba.

6. Orange-red indicates Ca.

7. Yellow, not seen through blue glass, indicates Na.

SOLUTIONS.

INDICATIONS OF COLORED SOLUTIONS.

Blue indicates copper.

Brown indicates copper-sulphides, or manganese.

Brown-yellow indicates ferric-salts, gold, platinum, or sulphides.

Brown-red indicates bromine.

Green indicates copper, nickel, chromium, or ferrous salts.

Purple-red indicates permanganates.

Yellow indicates neutral chromates.

Yellowish-red indicates acid chromates.

Red indicates cobalt, or manganese.

OXIDIZING AND REDUCING FLAMES.

When the Bunsen or alcohol flame is burning quietly, you will perceive a double cone of light, with a hollow center of unconsumed gas.

Do not allow the flame to burn too large.

Put the end of the blow-pipe in the center, and gently blow a non-luminous flame, or one nearly so. This flame contains oxygen in excess, highly heated, and tends to oxidize substances when they are placed in it. It is called the *oxidizing flame*.

By placing the end of the blow-pipe just outside the flame, and gently blowing, a flame somewhat luminous, and containing an excess of hydrogen and carbon at a high temperature, will be produced. This flame reduces or takes away oxygen from bodies placed in it, and is called the *reducing flame*.

When using the oxidizing flame, keep the substance for experiment within the flame of *complete* combustion at the end.

When using the reducing flame, keep the substance for experiment within the flame at the point of *incomplete* combustion.

BORAX-BEAD TEST.

Heat the platinum wire and dip it into powdered borax. Heat the borax in the flame. At first, the borax will puff up into a white spongy mass, then condense into a small glassy bead, which is to be dipped into the solution to be tested, and the flame again applied. Sometimes the coloring is slight unless the solution is somewhat concentrated.

The platinum wire for tests should be about three inches long, with one end fastened into a piece of small glass tubing for a handle, and the other end bent into the form of a small loop. After using it for tests, the wire should be thoroughly cleaned.

The wire is spoiled for tests when used with fusible metals, such as lead, antimony, or bismuth.

A thread of asbestos is preferred to platinum for the flame test.

OXIDIZING FLAME.		METAL.	REDUCING FLAME.
<i>Hot.</i>	<i>Cold.</i>		
Green.	Blue.	Cu.	Red tinge.
Blue.	Blue.	Co.	Blue.
Green.	Green.	Cr.	Green.
Red.	Yellow.	Fe.	Dark green.
Violet.	Brown.	Ni.	Gray.
Violet.	Amethyst.	Mn.	Nearly colorless.

BLOW-PIPE WITH CHARCOAL.

Place a small amount of the substance on charcoal and heat it in the reducing flame. No incrustation or metallic globule indicates salts of H_4N , or compounds of Hg; and when the odor of garlic is present, it shows As_4O_6 .

	Incrustation yellow, c—PbO.
Volatile with	“ dark yellow, c—Bi ₂ O ₃ .
incrustation.	“ yellow, h; dirty white, c—SnO ₂ .
Sometimes partially	“ white, c—Sb ₂ O ₃ .
volatile.	“ yellow, h; white, c—ZnO.
c = cold. h = hot.	“ reddish-brown—CdO.
	“ blue and yellow—MoO ₃ .
Fusible, non-volatile,	
absorbed, or	Indicates K, Na, Si, Ca, Sr, Ba.
forms a bead.	
Infusible, no change	SiO ₂ , Al ₂ O ₃ .
of color.	Some compounds of Mg, Sr, Ba, Ca.

CHAPTER IV.

CHEMICAL FORCE OF ELEMENTS.

VALENCE.

Valence has reference to the power of any atom to hold other simple atoms in combination. By referring to the table, it is seen to differ very much in different elements.

In determining valency, H is taken as the unit, and an element which can hold one atom of H only, is called an univalent element; one that can hold two atoms of H, or of any other univalent element, is called bivalent, and so on.

The valence of any element is determined by any univalent element with which it will enter into combination. Some elements have more than one valence.

Na and K, each being univalent, can only replace one atom of hydrogen.

HNO_3 (nitric acid) becomes KNO_3 (potassium nitrate) upon displacing its H by the univalent K. 2HNO_3 becomes $\text{Ca}(\text{NO}_3)_2$ (calcium nitrate) upon displacing its H by the bivalent Ca.

H_2SO_4 (sulphuric acid) becomes BaSO_4 (barium sulphate) upon displacing its H by the bivalent Ba.

A careful study of valence will assist in naming the results of chemical reactions. Valence is often referred to by other titles; as "quantivalence," "bonds of union," etc.

ATOMIC WEIGHT.

The **Atomic Weight** of an element is the weight of one of its atoms compared with that of an atom of hydrogen. The combining power of an element is proportional to its atomic weight. The molecular weight of a body is the sum of the atomic weights of the atoms of the various elements which make the compound. No matter how the compound is made, the proportion of elements by weight remains fixed.

This principle is used in determining the exact amount of an element in a compound.

EXAMPLE.

How many pounds of sodium (Na) in 25 pounds of salt (NaCl)?

23 (at. wt. Na) : 58.5 (molecular wt. NaCl) :: x : 25 lbs.

$$x = 9\frac{27}{117} \text{ lbs. Na.}$$

Rule. The atomic weight of the element to be considered is to the molecular weight of the compound as the weight of the element is to the weight of the compound.

An Atom is the smallest part into which an element can be divided. Most elements unite as atoms in forming compounds; a few unite as molecules.

A Molecule is the smallest portion into which matter can be divided without changing the nature of the substance.

Matter can be divided into its component molecules by the application of physical forces, but each molecule retains all the properties of the body which was divided. When, however, the molecule is separated into its component atoms, a chemical force is required, and the properties of the resulting atoms or molecules are different from those of the original body.—*G*.

Many elements in the free state have a molecule containing more than one atom, and the properties of the molecule differ from those of the atom. Thus the atom of hydrogen is H; the molecule of hydrogen in the free state is H_2 ; the atom of oxygen is O; the molecule of free oxygen is O_2 .

PROBLEMS UNDER ATOMIC WEIGHT.

1. How many grains of H are there in 50g. of H_2O ?
2. How much O is there in 8 lbs. of nitric acid?
3. How much O can be obtained from 1 lb. of $KClO_3$?
4. How much HNO_3 can be made from 75 lbs. of $NaNO_3$?
5. How much H_2O is there in 25 lbs. of sodium sulphate?
6. What weight of H can be obtained from 1 cu. ft. of distilled H_2O ?
7. How much CO_2 is formed from the burning of 100 lbs. of C?
8. How much S in 5 lbs. of sulphuric acid?
9. How much common salt is needed to make 25 lbs. of hydric chloride?
10. How much Fe in 20 lbs. of $FeSO_4$?
11. How much O in 12 lbs. of zinc sulphate?
12. How much $NiSO_4$ would be required to get 5 lbs. of nickel?
13. How much potassium in 30 lbs. of potassium nitrate?
14. How much sodium in 20 lbs. of baking soda?
15. How much $KClO_3$ would be required to obtain the K needed to make 20 lbs. of saleratus?

CHAPTER V.

PROCESS OF SOLUTION.

By reference to the Table of Solubility, it will be seen that some substances are soluble in water, some in acids, and others in neither water nor acids.

The process of making solutions is an important one. We first divide all substances into **metallic** and **non-metallic**, and then apply the proper process, as follows:

NON-METALLIC SUBSTANCES.

Process 1. *Soluble in water.* Reduce the substance to a powder, and place a small amount in water. If solution does not take place very soon, boil the water containing the substance. If complete solution does not take place upon boiling, decant and preserve the fluid for experiment, and treat the insoluble part by Process No. 2. If the substance shows no apparent diminution by the first process, make sure that it is insoluble by evaporating some of the fluid upon platinum foil; when no solution has taken place, there will be no residue upon the foil. The solution obtained is to be used for the determination of bases and acids. (See Process of Analysis, page 35.)

Process 2. *Insoluble in water but soluble in acids.* First treat the powdered substance with cold *dilute* hydrochloric acid, HCl. If it is not dissolved, slowly heat it to boiling, and boil for some time. If this does not dissolve

the substance wholly or partially, treat it in the same way with *strong* HCl. If the last operation secures the desired result, add the solution to the one in dilute acid, in order to make the test. If hydrochloric acid does not dissolve the solid, repeat the experiments last named, using HNO_3 in place of HCl. When these expedients fail, mix the two acids to form Aqua Regia, and try again. If the substance or some part of it still remains undissolved, then proceed to Process 3.

Note.—Keep a sharp look-out for indications. Sulphides, Cyanides, Sulphites, and Carbonates may produce effervescence. Chlorine odors may arise from the compounds of that element. The insoluble Cyanides may give rise to the odor of bitter almonds.

Process 3. *Insoluble substances* may be heated in a platinum crucible, or upon platinum foil, with 4 or 5 parts of Sodium Carbonate (Na_2CO_3), which will convert them into a soluble form, which may be treated as in Process 1 or Process 2. It is important to avoid using an excess of acid in making solutions.

Reducible metals, such as Sn, Bi, Sb, As, and Pb, should not be used with platinum, as a fusible alloy is produced that may destroy the vessel or foil.

The fused mass is generally treated with hot distilled water to remove the excess of Sodium Carbonate. Keep these washings for test purposes, as they contain the acid radical. Wash the precipitate thoroughly, and dissolve in HNO_3 or HCl. This solution can be analyzed for the basic substance.

Many substances which are insoluble in water and acids, are decomposed by continued boiling in alkaline carbonates.

METALLIC SUBSTANCES.

Treat the substance with Nitric Acid, applying heat if necessary. It may be a simple metal or an alloy.

a. If not acted upon by Nitric Acid, gold, platinum, aluminum, or iridium are present.

b. Antimony and tin form oxides, which do not dissolve in an excess of water or the acid.

c. Other metals are converted into *nitrates*, which are soluble in an excess of water or the acid.

d. Complete solution in HNO_3 or H_2O shows an absence of the metals named in a and b.

e. When there is a residue, and it is metallic, first treat it with water and then with hydrochloric acid. Dissolving with effervescence indicates Aluminum. Then try with Aqua Regia; if any residue is left, apply gentle heat, and examine the solution for gold, platinum, or iridium.

f. If the residue is a white, powdery substance, antimony or tin may be present. Pour off the solution, wash the residue with water, dissolve the powder in HCl , and apply the tests for those metals. (See p. 53.)

Note a.—We use the acids in the following order: HCl , HNO_3 , and Aqua Regia, first in dilute form, then in the concentrated form; first cold, and then hot.

Note b.—The solution should be neutralized, or the excess of acid removed, by adding sodium carbonate until (boiling or) effervescence ceases, or until the red and blue litmus papers are not wholly changed in color when both are added to the solution.

Note c.—It is usually better first to evaporate nearly all of the acid fluid, then dilute with water, after which add the sodium carbonate.

Note d.—If there is an unknown acid in the acid solution, it must be determined by tests before neutralizing. The presence of an acid is determined by using the litmus test.

Note e.—Sometimes the solution is strongly alkaline, determined by the litmus test, when it should be neutralized by carefully adding acetic or hydrochloric acid.

Note f.—Sometimes, to avoid too great a dilution, it is best to add the sodium carbonate in the dry form. The usual way is to add it in the form of a solution in water.

Note g.—Before using filter paper, it is better to dip it into distilled water, as this prevents the solution being absorbed by the paper.

CHAPTER VI.

PROCESS OF ANALYSIS.

GROUP REAGENTS.

The analysis of unknown solutions should be conducted upon some methodical plan. "In this study, heedless haste and slovenly manipulation mature an early harvest of error and disappointment."

The metals are divided into Five Groups, the members of each group being separated from the solution in succession, by the corresponding reagent. There are Four Group Reagents: HCl , which precipitates the members of the First Group as chlorides; H_2S , which precipitates the members of the Second and Third Groups as sulphides; $(\text{H}_4\text{N})\text{HS}$ or $(\text{H}_4\text{N})_2\text{S}_2$, which dissolves the sulphides of the Third Group, leaving those of the Second; and $(\text{H}_4\text{N})_2\text{CO}_3$, which precipitates the members of the Fourth Group as carbonates. The Fifth Group of metals is composed of those which are not precipitated by either of the above reagents.

Many analysts prefer to use sodium sulphide, Na_2S , or hydrogen sodium sulphide, NaHS , to dissolve the sulphides of the Third Group in place of $(\text{H}_4\text{N})_2\text{S}_2$. The subsequent steps of the process are precisely the same.—*G*.

The determination of the *bases* is the first step in analysis.

SEPARATION OF METALS OF GROUP ONE.

A. 1. Suppose an unknown solution is before us for analysis. We place a portion of it in a test tube, and cautiously add dilute HCl. If a precipitate forms, continue to add the acid until no more precipitate is thrown down. The precipitate will contain metals in the form of *chlorides*, which may be Pb, Ag, or Hg, one or all.

The precipitates are all white. The chloride of each of these metals must next be separated from the mixture.

2. Arrange a filter in a glass funnel, and turn the contents of the test tube upon the filter. When the filtrate has all passed through, wash the precipitate with cold distilled water. Usually, it is not a good plan to let the washings run into the filtrate as it may dilute it too much. Preserve the filtrate (that which runs through) to be tested by the Second Group reagent. (See **B**, p. 37.)

3. Pour some boiling water upon the washed precipitate. This dissolves the PbCl_2 , which is soluble in hot water, though not in cold, but leaves the other chlorides. Repeat the washing two or three times to make sure that the PbCl_2 is all dissolved. Divide the solution of PbCl_2 into three parts and test each for Pb by the first, third, and fifth tests. (See tests for lead, p. 50.)

4. Pour warm $(\text{H}_4\text{N})\text{HO}$ upon the residue in the filter. This dissolves AgCl and causes it to pass into the filtrate; at the same time, the HgCl is blackened by being turned into $\text{NH}_2\text{Hg}_2\text{Cl}$.

5. Add HNO_3 to the filtrate which contains the AgCl until the $(\text{H}_4\text{N})\text{HO}$ is neutralized, when the AgCl is reprecipitated. Filter out the AgCl , spread the filter and

expose it to the sunlight; the presence of Ag is confirmed by the blackening of the precipitate.

When there is considerable of the AgCl, the presence of Ag can be still further proved by drying a portion, mixing it with sodium carbonate, Na_2CO_3 , and heating it on charcoal before the blowpipe in the reducing flame; metallic Ag is produced.—*G*.

6. The black precipitate still remaining in the filter is to be tested for Hg. This may be done by dissolving it in Aqua Regia and applying either of the special tests. (See p. 51.)

A very convenient method for testing the Hg, and one which all students should apply, is the following: Evaporate the solution in Aqua Regia to a small amount, add a little water and filter. This removes any AgCl which failed to be dissolved. Then place a piece of clean copper wire in the filtrate, and boil; the copper turns gray from precipitated Hg.

By heating the wire in a closed tube, the Hg is sublimed and appears as minute drops upon the side of the tube.—*G*.

Note.—The *mercurous* salts alone belong to this First Group; the *mercuric* salts belong to the Second Group. Lead is incompletely precipitated in the First Group, but is completely precipitated in the Second Group.

SEPARATION OF METALS OF GROUP TWO.

B. 1. To the filtrate from the First Group, **A**, add cautiously H_2S . If a precipitate appears, continue to add H_2S until no more precipitate is thrown down.

2. The precipitate will contain metals in the form of *sulphides*, which may be Pb, As, Sb, Sn, Bi, Cu, Cd, Hg, Au, and Pt; also some rarer metals, one or all.

3. The sulphides are colored as follows, viz.: As—yellow; Sb—orange; Sn—brown or yellow, brown indicat-

ing stannous, yellow indicating stannic salts; Pb, Bi, Cu, and Hg are black; Cd is yellow.

4. It is useless to search for Pb or Cu in a light colored precipitate; but we must not suppose or infer that the precipitates of the lighter colors are absent from the black. Their presence or absence must be determined by careful tests.

5. Thoroughly wash the precipitate as in First Group, and preserve the filtrate for examination for the Third Group. (See **C**, p. 40.)

6. The precipitates consisting of the mixed sulphides are treated with $(\text{H}_4\text{N})_2\text{S}_2$, yellow ammonium sulphide, which dissolves some of the Second Group sulphides, and forms the First and Second Parts.

Many analysts prefer to use Na_2S , sodium sulphide, to make this separation.—*G*.

First Part.

This is the remaining precipitate, and contains one or more of the sulphides of Hg, Pb, Bi, Cu, and Cd, which make Group Two.

Second Part.

This is the filtrate, and contains one or more of the sulphides of Sn, Sb, As, and rarely Au and Pt, which make Group Three.

Note.—In using the ammonium sulphide for this separation, use it in an evaporating dish, and digest for some time with gentle heat.

To remove the precipitate from the filter to the evaporating dish, the filter may be spread out upon a clean glass plate and the precipitate scraped off with a horn or ivory spatula; or, a hole may be made in the bottom of the filter as it rests in the funnel, and the precipitate washed down with a fine stream of water from the wash bottle.—*G*.

Separation of Metals of First Part.

1. Boil the sulphides in dilute HNO_3 . A black insoluble residue indicates HgS .

2. Treat the residue with Aqua Regia, boil down to expel the excess of acid, and test for Hg with copper wire, as in Group One, and with SnCl_2 . (See tests for Hg, p. 51.)

3. Test the filtrate for Pb by adding H_2SO_4 to a small portion. If a precipitate forms, add H_2SO_4 to the entire filtrate, which will remove the lead as a white precipitate of lead sulphate, PbSO_4 . Filter, and proceed with the filtrate to ascertain the presence of Bi, Cu, and Cd.

4. To the last filtrate, add an excess of ammonia. Bi is precipitated as bismuthous oxide, Bi_2O_3 .

To confirm its presence, the Bi_2O_3 is filtered out and dissolved in a small amount of HCl . The solution is then evaporated to one or two drops and poured into a test tube containing cold water; a white precipitate of BiOCl , Bismuth oxy-chloride, is formed.—*G*.

5. Cu and Cd, if present, remain in the solution. The presence of Cu tinges the solution a blue color.

6. To separate Cu and Cd, add H_2S to the solution, which precipitates both of them. Add hot dilute H_2SO_4 to the precipitate on the filter paper; Cd is placed in solution and passes into the filtrate.

The presence of copper can be confirmed by again dissolving the CuS in $(\text{H}_4\text{N})\text{HO}$ and applying the tests for the metal. (See p. 54.) The solution can be tested for Cd by neutralizing the H_2SO_4 with $(\text{H}_4\text{N})\text{HO}$ and applying the tests for the metal. (See p. 54.)—*G*.

Separation of Metals of Second Part.

1. The sulphides of Sn, Sb, and As are precipitated from the filtrate by adding HCl. Filter, and wash the precipitate with hot water.

2. Place the precipitate in a flask, and boil with concentrated HCl until you can not detect the odor of H_2S , and a piece of filter paper moistened with a solution of lead acetate is not darkened

3. Sb and Sn are dissolved, while As_2S_3 remains undissolved. Filter and wash. Boil the residue in strong HCl with a few crystals of KClO_3 . As_2S_3 is converted into H_3AsO_4 , arsenic acid, which may then be tested. (See tests for As, p. 52.)

4. To the last filtrate, add Zn and a piece of platinum foil, bringing the Zn into contact with it. Sn and Sb, if present, are reduced to the metallic state. Sn is deposited on the Zn, and Sb on the Pt, as a black stain. Au and Pt being insoluble in HCl, remain with the residue containing As.

5. Treating the precipitate with KClO_3 and HCl, Au and Pt are dissolved.

SEPARATION OF METALS OF GROUP THREE.

C. 1. The filtrate from the Second Group, **B**, should be boiled to expel H_2S .

2. After adding a little HNO_3 , boil for a time to change ferrous salts to ferric, if any are present.

3. Add H_3N and H_4NCl . Fe, Cr, and Al are precipitated as oxides.

4. To the filtrate, add $(\text{H}_4\text{N})\text{HS}$. Ni, Co, Mn, and Zn are precipitated as sulphides.

5. Boil the precipitate containing Fe, Cr, or Al for several minutes in KHO . The aluminum is dissolved. Filter and wash.

6. Dissolve a small portion of the precipitate in HCl , which turns the oxide to the chloride, and test it for Fe. (See tests for Fe, p. 54.)

7. Fuse a second part of the precipitate on platinum foil with KNO_3 and Na_2CO_3 ; boil the fused mass in water, and then test it for Cr. (See tests for Cr, p. 55.)

Boil the remainder of the precipitate in an excess of NaHO , sodium hydrate. This dissolves the Al and turns it to $\text{Na}_2\text{Al}_2\text{O}_4$, sodium aluminate. Filter, and add HCl to the filtrate until it produces acid reaction. This produces Al_2Cl_3 , aluminum chloride. Add H_4NHO , and a flocculent precipitate of $\text{Al}_2\text{H}_6\text{O}_6$, aluminum hydrate, is produced.—*G*.

8. The color of the precipitates is as follows, viz.: Fe, reddish brown; Al, colorless and gelatinous; Cr, bluish-green; Mn, flesh color; Ni and Co, black; Zn, white.

9. If the precipitated sulphides of one or more of the following metals—viz.: Ni, Co, Mn, or Zn—be light colored, cobalt and nickel must be absent; if dark, one or both are present.

10. If dark colored, treat it with cold HCl , and then filter. The filtrate will contain the Mn and Zn, if present.

11. Test the precipitate for Co with the borax-bead. (See Borax-Bead Test, p. 26.)

12. If Co is present, dissolve the precipitate in a small amount of Aqua Regia, and add NaHO until the precipitate

is permanently formed. Add acetic acid and a strong solution of KNO_2 , potassium nitrite. Warm the solution gently, or permit it to stand in a warm place for about twenty hours, and the Co will be precipitated.

13. Filter; the filtrate will contain the Ni, whose presence may be confirmed by the tests for that metal. (See tests for Ni, p. 55.)

14. Boil the filtrate containing Mn and Zn, to expel H_2S , and add an excess of either NaHO or KHO . After standing for a little time, Mn is precipitated as Mn_2HO , which is white. (See tests for Mn, p. 56.) Filter and test the filtrate for Zn by the second and third tests. (See tests for Zn, p. 56.)

SEPARATION OF THE METALS OF GROUP FOUR.

D. 1. Boil the filtrate from the Third Group, C, to expel the H_2S .

2. If the solution be acid, render it alkaline by adding $(\text{H}_4\text{N})\text{HO}$.

3. Add a small amount of H_4NCl , warm the mixture, and then add $(\text{H}_4\text{N})_2\text{Co}_3$ as long as a precipitate is formed.

4. The precipitate may contain Ba, Ca, and Sr, one or all, in the form of white carbonates. Filter, and wash the precipitates thoroughly with hot water while they are on the filter.

5. Dissolve the precipitate in acetic acid. Test for Ba by adding either $\text{K}_2\text{Cr}_2\text{O}_7$ or K_2CrO_4 to a small portion. If Ba is present, a yellow precipitate, BaCrO_4 , is formed. If Ba is present, remove it from the whole solution, filter

and dissolve the residue in HCl, and apply further tests for Ba. (See tests for Ba, p. 56.)

6. To the filtrate which may contain Sr or Ca, add its own volume of K_2SO_4 , and the strontium is precipitated as $SrSO_4$, strontium sulphate. Some time should be given for the precipitate to form.

7. To the filtrate obtained by removing Sr, add H_3N and ammonium oxalate, $(H_4N)_2C_2O_4$. This gives a white precipitate insoluble in acetic acid but soluble in HCl, which is distinctive of Ca.

Notes by La Roy F. Griffin.

Another process for separating these metals, which is somewhat more accurate than the preceding, is as follows:

Filter out the precipitated carbonates, and wash the precipitate into a test tube, add a small quantity of HCl: it changes the carbonates to chlorides.

Evaporate the solution, cautiously, to dryness, in a small evaporating dish. A crystalline residue remains, which is then treated with $(C_2H_5)HO$, ethylic alcohol. The crystals should be pulverized carefully with the end of a stirring rod that the $(C_2H_5)HO$ may act upon every part: the $SrCl_2$ and $CaCl_2$ are dissolved, while the $BaCl_2$ remains. (It is well to repeat this process to render the separation complete.)

The residue may now be tested for Ba. (See tests for Ba, p. 56.)

Add dilute H_2SO_4 to the filtrate. This produces a white, gelatinous precipitate of mixed strontium sulphate, $SrSO_4$, and calcium sulphate, $CaSO_4$, with a trace of $BaSO_4$, which must be removed to make the final tests reliable. Filter out the precipitate, transfer it to an evaporating dish, and boil it for five minutes, in a mixture of sodium carbonate, Na_2CO_3 , and potassium sulphate, K_2SO_4 . This turns the $SrSO_4$ and $CaSO_4$ to carbonates, while the $BaSO_4$ remains, but soon dissolves. Filter; the precipitate in the filter consists of mixed $SrCO_3$, and $CaCO_3$. Wash it thoroughly with water.

Now, transfer the precipitate to a test tube, and dissolve it in HCl; the effervescence is caused by the escape of CO_2 . Evaporate the solution nearly to dryness to expel all HCl, and dissolve it in water. This solution consists of SrCl_2 and CaCl_2 .

If the solution is not clear, filter out the impurities.

Divide the solution into two parts, and test one for Sr by adding CaSO_4 . A white precipitate of SrSO_4 shows the presence of Sr.

Test the other for Ca by adding potassium sulphate K_2SO_4 , to remove the Sr as SrSO_4 . Filter, and add ammonium oxalate, $(\text{H}_4\text{N})_2\text{C}_2\text{O}_4$, to the filtrate. A white precipitate of calcium oxalate, CaC_2O_4 , shows the presence of Ca.

Sometimes this precipitate does not appear until after standing for some time. Consequently it is well to allow it to stand for twenty-four hours before deciding that Ca is absent.

SEPARATION OF THE METALS OF GROUP FIVE.

E. The filtrate from Group Four (see **D**, No. 4) may contain the metals of Group Five, Mg, H_4N , K, and Na. The solution is divided into *three parts*.

1. *The first part* is to be tested for Mg by adding $(\text{H}_4\text{N})\text{HO}$ to alkaline reaction, as shown by turning red litmus paper blue, and then di-sodium phosphate, Na_2HPO_4 . This forms a white precipitate of ammonio-magnesium phosphate, H_4NMgPO_4 . Frequently, it only appears after the mixture has stood for some time.

2. *The second part* is to be evaporated almost to dryness, and tested for Na and K by the Flame Test. (See Flame Test, p. 24.) Their presence may be confirmed by the special tests for those metals. (See p. 57.)

3. *The third part* is to be tested for H_4N by adding NaHO (See tests for H_4N , p. 57.)

Note.—As ammonium has been added in the process of testing for members of the other groups, it will be found in this filtrate. Therefore test for H_4N in a portion of the original solution.

CHAPTER VII.

PRELIMINARY STEPS.

The First Step is to place a small amount of the powdered substance in a dry test-tube—a hard glass or ignition tube is best—and apply a gentle heat at first, then full heat, carefully noting the changes.

Laboratory students can use a closed tube made over the lamp flame from glass tubing. The most convenient method is to cut off a piece of tubing about five inches long, and melt the middle, drawing it out as it melts, and so close both pieces. A new tube can be used for each test. The following reactions are to be noted, and any indication should be followed by a test for the substance indicated.—*G.*

A. *If the substance remains unchanged*, it shows the absence of volatile, fusible or organic matter; also, the absence of water of crystallization, and substances whose color is changed by heat.

B. *If the substance changes color*, it may be from the forming of an oxide, or the charring of organic matter. Smoky fumes usually arise from the charring of organic matter. The residue shows the following indications or changes. The first color is for the substance while hot, the latter color when it has become cold.

Bi_2O_3 ,	red-brown	to pale yellow.
PbO ,	red-brown	to yellow.
Fe_2O_3 ,	black	to red.
ZnO ,	yellow	to white.
SnO_2 ,	yellow-brown	to yellow.
Co and Cu,	black, after intense heating.	

C. *A sublimate is formed* (*sublimation* meaning the changing into vapor by heat, and condensing again when cooled).

Hg is indicated by a gray tarnish or distinct globules.

Hg Cl sublimes without fusion; yellow while hot, white when cold.

HgCl₂ melts, then sublimes, showing crystals, white when cold.

A white sublimation without fusion, giving ammonia odors, changing red litmus to blue when heated with sodium carbonate, indicates salts of H₄N.

As₄O₆ produces a white sublimate, no fusion, octahedral crystals seen through a lens.

Sb₂O₃ fuses and sublimes in needle-shaped crystals.

HgS gives a black sublimate, which becomes red when rubbed with a glass rod.

S sublimes in reddish-brown drops.

D. *If watery vapor is driven off*, it may come from water of crystallization, or water held mechanically, or some decomposable hydrates, as borax.

E. *Gases or fumes driven off upon the application of heat.*

O₂, tested by plunging into it a glowing splinter, comes from the bromates, chlorates, iodates, nitrates, and peroxides.

CO₂, tested by lime water, with which it produces a white precipitate of CaCO₃, calcium carbonate, comes from the carbonates.

SO₂, known by the odor, comes from the compounds of sulphur.

H_2S comes from the sulphides containing water, and is known by the odor of rotten eggs.

H_3N is given off from the salts of ammonia. The tests are moistened red litmus paper, which is blue, or moistened turmeric paper, which is turned brown, and bringing the gas into contact with HCl gas, in which case, white fumes are formed.

CN , known by its odor, is given off by certain cyanides.

NO_2 produces reddish-brown fumes, from the decomposition of the nitrates.

CO burns with a blue flame, and indicates the presence of oxalates.

Cl , Br , and I , are recognized by their odor and color, and show the presence of chlorides, bromides, or iodides.

The Second Step is to apply the **Flame Test**, noting the colors carefully. (See **Flame Test**, p. 24.)

The Third Step is to apply the **Charcoal Test**. (See "**Blow-pipe with Charcoal**," p. 26.)

The Fourth Step is to give the **Test with Borax**. (See "**Borax-Bead Test**," p. 26.)

The Fifth Step is to mix the substance with a small amount of Na_2CO_3 , and heat in the reducing flame on charcoal.

The amount of the Na_2CO_3 should be about double that of the substance to be tested.

A mixture of Na_2CO_3 and KCy makes a powerful reducing agent, which becomes necessary in treating the compounds of tin.

A. Metallic globules with no coating or incrustation.

Cu gives a red, malleable globule.

Au gives a yellow, malleable globule.

Ag gives a silvery-white, malleable globule.

B. *Metallic particles, powder or grains with no incrustation.*

Fe, Ni, Co, are magnetic and infusible.

Pt, Ir, are non-magnetic and infusible.

C. *Incrustation without metallic particles or powder.*

As, odor of garlic, very volatile, white.

Cd, reddish-brown and volatile.

Zn, yellow while hot, white when cool.

D. *Metallic particles or globules with incrustation.*

Sb, white coating, globule easily formed, hard and brittle.

Pb, yellow coating, globule easily formed, soft and malleable.

Bi, yellow coating, globule easily formed, fusible and brittle.

Sn, pale yellow, to white on cooling, globule formed with difficulty, malleable and fusible.

The Sixth Step is to heat the substance on charcoal in the reducing flame, then remove and moisten with a small amount of cobalt-nitrate, $\text{Co}(\text{NO}_3)_2$, then return to flame, and heat intensely.

A colored mass will be given, the color of which will be observed when *cold*.

ZnO shows yellowish-green.

Sb_2O_3 shows dingy-green.

SnO_2 shows bluish-green.

MgO shows pink.

Al_2O_3 shows blue.

SiO_2 shows pale blue.

The alkaline silicates, phosphates, and borates give a bead of blue glass.

The Seventh Step, if the substance be in a liquid form, is to test with the litmus papers to ascertain whether it be acid or alkaline. Place some of the liquid on platinum foil, and evaporate to ascertain if there be any residue.

The Eighth Step is to use the Group Reagents and proceed with the precipitates as indicated in Chapters VI. and VIII.

CHAPTER VIII.

TESTS FOR THE METALS.

METALS OF THE FIRST GROUP.

Note 1.—pp. stands for *precipitate* in the following tests.

Note 2.—In the following tests it is supposed that the solution from the preceding Group Reactions, which contains the metal to be tested, is at hand.

Note 3.—Any of the common salts can be put into solutions and the tests applied without using the Group Reagents.

Lead (Pb).

1. Add H_2SO_4 , white pp. (PbSO_4), insoluble in acids.
2. “ $(\text{H}_4\text{N})_2\text{CO}_3$, white pp. (PbCO_3).
3. “ $\text{K}_2\text{Cr}_2\text{O}_7$, yellow pp. (PbCrO_4).
4. “ KI , yellow pp. (PbI_2), soluble in H_2O .
5. “ H_2S , black pp. (PbS), soluble in hot HNO_3 .
6. “ HCl , white pp. (PbCl_2), soluble in boiling H_2O .
7. “ NaHO , white pp. (PbH_2O), soluble in excess of NaHO .

Silver (Ag).

1. Add HCl , white pp. (AgCl), soluble in H_3N and not in HNO_3 .
2. Add H_2S , black pp. (Ag_2S), insoluble in alkalies.
3. “ KHO , brown pp. (Ag_2O).
4. “ KI , yellowish-white pp. (AgI), insoluble in acids, slightly soluble in H_3N .

5. Add KCy, white pp. (AgCy).
6. “ K_2CrO_4 , dark-red pp. (Ag_2CrO_4), soluble in HNO_3 .
7. “ Na_2HPO_4 , yellow pp. (Ag_3PO_4).

Mercury (Hg).**Mercurous Salts.**

1. Add H_2S , or $(H_4N)_2S$, black pp. (Hg_2S), insoluble in hot HNO_3 , soluble in Aqua Regia.
2. Add NaHO, black pp. (Hg_2O).
3. “ KI, yellowish-green pp. (HgI).
4. “ $SnCl_2$, gray-metallic pp. (Hg).
5. “ HCl , white pp. ($Hg Cl$). Filter; and add H_3N ; turns black.

Note.—See Hg (mercuric conditions) in Second Group.

METALS OF THE SECOND GROUP.**Mercury (Hg).****Mercuric Salts.***(See First Group.)*

1. Add NaHO, yellow pp. (HgO).
2. “ K_2CrO_4 , orange pp., soluble in HNO_3 .
3. “ $SnCl_2$, white pp. ($Hg Cl$). If added in excess gray metallic Hg is deposited.
4. Add KI, yellow pp. (HgI_2), rapidly becoming scarlet; soluble in an excess of KI or $HgCl_2$.
5. Add H_3N , white pp. (H_2NHgCl).
6. Add H_2S , white pp. changing to orange, brown, and black (HgS); insoluble in hot HNO_3 , soluble in Aqua Regia.

7. *The Copper Test.* Place a small piece of bright copper, about one-half inch long and one-fourth inch broad, in a solution of any salt of mercury, and heat in a test tube. The copper becomes coated with mercury in a

fine state of division. Avoid an excess of HNO_3 , or the Cu will be dissolved. To dispose of HNO_3 , add an alkali until a slight precipitate appears, then add a few drops of acetic acid or HCl .

After the Cu has been coated with the Hg, the test can be confirmed by putting the piece into a closed tube and heating. The Hg is driven off, and collects in minute drops on the side of the tube.

Arsenic (As).

Add H_2S , yellow pp. (As_2S_3), soluble in NaHO .

Note.—There are two classes of As salts—viz.: As_4O_6 , *Arsenious*, and As_2O_5 , *Arsenic*.

Arsenious.

1. Add H_2S , bright yellow pp. (As_2S_3), readily soluble in $(\text{H}_4\text{N})_2\text{CO}_3$.

2. Dissolve in HCl ; add a piece of clean copper; a gray coating is found.

3. Add AgNO_3 to a neutral solution, yellow pp. (Ag_3AsO_3).

4. Add Ammonio-Sulphate of Copper, green pp. (CuHAsO_3), *Scheele's Green*, soluble in HCl and H_4NHO .

5. Add Ammonio-Nitrate of Silver, yellow pp. (Ag_3AsO_3), easily soluble in HNO_3 and in H_4NHO .

6. Take arsenic green from wall paper, moisten with H_3N , place solution on porcelain, a yellow pp. will be formed by adding a crystal of AgNO_3 .

Arsenic.

1. Add AgNO_3 to a neutral solution, reddish-brown pp. (Ag_3AsO_4).

2. Add Ammonio-Nitrate of Silver, brick-red pp. (Ag_3AsO_4), soluble in HNO_3 and in H_4NHO .

3. Add Ammonio-Sulphate of Copper, light greenish-blue pp. (Cu_3AsO_4).

Note.—See Marsh's Test for Arsenic, in Appendix.

Antimony (Sb).

1. Add H_2S , orange pp. (Sb_2S_3), soluble in NaHO , insoluble in $(\text{H}_4\text{N})_2\text{CO}_3$.

2. Add H_4NHO , white pp. (SbH_3O_3).

Note.—See Marsh's Test for Arsenic, in the Appendix, and read distinction between As and Sb.

Tin (Sn).

Stannous Salts.

1. Add H_2S , dark brown pp. (SnS), soluble in yellow $(\text{H}_4\text{N})_2\text{S}_2$, re-precipitated by HCl , yellow pp. (SnS_2).

2. Add HgCl_2 , white pp. (HgCl).

3. “ H_4NHO , white pp. (Sn_2HO).

4. “ KHO , white pp. (Sn_2HO), dissolves in excess of KHO . Boil the solution, Sn is deposited as black pp. (SnO).

Stannic Salts.

1. No pp. occurs in No. 4 above, in stannic salts.

2. Add H_2S , yellow pp. (SnS_2), soluble as in No. 1 above.

Bismuth (Bi).

Note.—If you use $\text{Bi}(\text{NO}_3)_3$ for test solution, boil it in solution with water and the pp. will fall; turn off the clear fluid and apply the tests.

1. Add KHO , white pp.

2. “ H_2O , white pp., insoluble in tartaric acid.

3. “ H_2S , black pp. (Bi_2S_3), soluble in HNO_3 .

4. Add H_4NHO , white pp. (BiHO_3).
5. “ $\text{K}_2\text{Cr}_2\text{O}_7$, yellow pp. ($\text{BiO}_2\text{Cr}_2\text{O}_7$), insoluble in KHO .
6. Add H_4NHO , white pp. (BiOHO), becomes yellow on boiling.

Copper (Cu).

1. Add H_2S , black pp. (CuS), soluble in HNO_3 .
2. “ H_4NHO , greenish-blue pp. (Cu_2HO).
3. “ $(\text{H}_4\text{N})\text{HS}$, purplish red or dark pp. (CuS).
4. “ KHO , green pp. (Cu_2HO), turns black by boiling.
5. Add K_4FeCy_6 , reddish-brown pp. (Cu_2FeCy_6).
6. “ KC_y , greenish-yellow pp. (CuCy_2), soluble in excess.

Gold (Au).

1. Add H_2S , brown pp. (Au_2S_3).
2. “ Fe_2Cl_6 , set aside, metallic Au is precipitated.
3. “ SnCl_2 to a drop of Au solution on white paper, a rich purple will appear. *Purple of Cassius.*
4. Boil some of the solution of Au in oxalic acid. The Au is precipitated in metallic form.

*NH₄Cl***Cadmium (Cd.)**

1. Add H_4NHO , white pp. (Cd_2HO), soluble in excess.
2. “ H_2S , yellow pp. (CdS), soluble in HNO_3 , insoluble in KC_y .
3. Add KC_y , white pp. (Cd_2Cy), soluble in excess.

METALS OF THE THIRD GROUP.**Iron (Fe).****Ferrous Salts.**

1. Add K_4FeCy_6 , white pp. ($\text{K}_2\text{Fe}_2\text{Cy}_6$), rapidly becomes blue.

2. Add $(\text{H}_4\text{N})\text{HS}$, black pp. (FeS).
3. “ K_6Fdcy (ferricyanide of potassium), blue pp. (Fe_3Fdcy), Turnbull's blue, is thrown down.
4. Add $(\text{H}_4\text{N})_2\text{CO}_3$, white pp. (FeCO_3), quickly turns dark.

Ferric Salts.

1. Add K_4FeCy_6 , a blue pp., ($\text{Fe}_4\text{Fe}_3\text{Cy}_{18}$), Prussian blue.
2. Add $(\text{H}_4\text{N})_2\text{CO}_3$, reddish brown pp. (Fe_26HO).
3. Same as 2, ferrous salts.
4. Add H_3N , reddish-brown pp. ($\text{Fe}2\text{HO}$).
5. “ Nut-gall, blue-black pp.

Chromium (Cr).

1. Add H_2S , changes color to green.
2. “ Lead acetate, yellow pp. (PbCrO_4).
3. “ AgNO_3 , brownish-red pp. (Ag_2CrO_4).
4. “ $(\text{H}_4\text{N})_2\text{S}$, bluish-green pp. (Cr_26HO), soluble in acids.

Aluminum (Al).

1. Add Na_2HPO_4 , white pp. $\text{Al}_2(\text{PO}_4)_2$, soluble in KHO , insoluble in acetic acid.
2. Add $(\text{H}_4\text{N})_2\text{S}$, white pp. (Al_26HO), flocculent.

Nickel (Ni).

1. Add $(\text{H}_4\text{N})\text{HS}$, black pp. (NiS).
2. “ KHO , light green pp. ($\text{Ni}2\text{HO}$).
3. “ H_4NHO , greenish or faint-blue pp. ($\text{Ni}2\text{HO}$), excess of H_4NHO dissolves pp. in 2 and 3.

Cobalt (Co.)

1. Add H_4NHO , blue pp. ($\text{Co}2\text{HO}$), dissolves in excess of H_4NHO .

2. Add $(\text{H}_4\text{N})\text{HS}$, black pp. (CoS) , insoluble in HCl , soluble in HNO_3 .

3. Add KC_y , brownish-white pp. $(\text{CoC}_y)_2$, soluble in excess of KC_y .

Manganese (Mn).

1. Add $(\text{H}_4\text{N})\text{HS}$, flesh colored pp. (MnS) , turns to brown.

2. Add KHO , white pp. (Mn_2HO) , turns brown by shaking.

3. Add H_4NHO , white pp. (Mn_2HO) , turns brown.

Zinc (Zn).

1. Add $(\text{H}_4\text{N})\text{HS}$, white pp. (ZnS) , insoluble in acetic acid, soluble in HNO_3 .

2. H_4NHO gives white pp. (Zn_2HO) , soluble in excess; given again by boiling.

METALS OF THE FOURTH GROUP.

Barium (Ba).

1. Add $\text{K}_2\text{Cr}_2\text{O}_7$ and H_4NHO , light yellow pp. (BaCrO_4) , insoluble in acetic acid. Without H_4NHO deep yellow pp. is given.

2. Add $(\text{H}_4\text{N})_2\text{CO}_3$, white pp. (BaCO_3) .

3. “ weak H_2SO_4 , white pp. (BaSO_4) , insoluble in acids.

4. Add K_2CrO_4 , light yellow pp. (BaCrO_4) .

5. “ Na_2HPO_4 , white pp. (BaHPO_4) .

Strontium (Sr).

1. Add $(\text{H}_4\text{N})_2\text{CO}_3$, white pp. (SrCO_3) .

2. “ Na_2CO_3 , white pp. (SrCO_3) .

3. Add $(\text{H}_4\text{N})_2\text{C}_2\text{O}_4$ white pp. (SrC_2O_4), soluble in HCl .

Calcium (Ca).

1. Add $(\text{H}_4\text{N})_2\text{CO}_3$, white pp. (CaCO_3).

2. “ $(\text{H}_4\text{N})_2\text{C}_2\text{O}_4$, white pp. (CaC_2O_4), soluble in HCl .

Magnesium (Mg).

1. Add H_4NHO and Na_2PHO_4 , faint white pp. (MgH_4NPO_4).

2. Add $(\text{H}_4\text{N})_2\text{CO}_3$, white pp. (MgCO_3), when boiled soluble in excess of $(\text{H}_4\text{N})\text{Cl}$.

METALS OF THE FIFTH GROUP.

Potassium (K).

1. Add tartaric acid, strong solution, white pp. acid tartrate of potassium ($\text{KH}_5\text{C}_4\text{O}_6$) is formed by stirring. Solution must be cold and somewhat concentrated.

2. Add PtCl_4 , yellow pp. ($\text{PtCl}_4 \cdot 2\text{KCl}$) is formed by stirring.

Sodium (Na).

1. Add H_2SiF_6 , white pp. (Na_2SiF_6), gelatinous.

Ammonium (H_4N).

1. Add KHO and heat, gaseous H_3N escapes, and may be detected by the sense of smell.

2. A warm glass rod wet with HCl , and brought near fumes in 1 will give white fumes of (H_4NCl).

3. Add PtCl_4 , yellow pp. ($\text{PtCl}_4 \cdot 2\text{H}_4\text{NCl}$), insoluble in alcohol. (See test for H_4N , in Appendix.)

Note.—A large number of the metals should be tested by the *flame*, *borax-bead*, or *charcoal*.

CHAPTER IX.

TESTS FOR ACIDS.

PRELIMINARY TESTS.

Note.—The solution for analysis should be divided into two or more portions, one of which is to be used in testing for *bases*, and the other is to be used in testing for *acids*.

1. There are no methods of grouping the *acids* in a definite way, as we have done with the *bases*; yet certain steps can be taken that will indicate in general the plan to be pursued.

2. Since, in searching for metals, acids have been introduced into the solution, we must not use the filtrate left from which to determine the acids originally present, but we must apply our tests to a portion of the original solution.

3. We must keep carefully in mind the kinds of materials we use in the process of making solutions, as their presence will be certain in the solution.

4. After finding the *basic* element, the Table of Solubilities will render important service, in showing the probable absence or presence of some *acids*.

It will be seen readily that it would be of no use to seek in a solution for an acid whose salt with the metal previously determined, is insoluble in water, in acids, or in a mixture of water and acids. For example, you would not look for phosphoric acid, H_3PO_4 , with iron, as they make

a compound which is insoluble in water. At the same time, Fe might be found with H_2SO_4 in H_2O .

5. The *bases* of Groups from 1 to 4 inclusive, may be removed from the solution by boiling with Na_2CO_3 , filtering, and adding HNO_3 to the filtrate, merely enough of the latter to make it neutral, and then boiling to expel CO_2 . Some of the sodium salts will contain the *acids* of the solution.

6. The metals of the alkalies (K, Na, H_4N), may be in the filtrate from Group 5, but they can be determined by the tests previously mentioned.

7. The removal of the *bases*, as a rule, should be insisted upon, as their presence may interfere with the detection of some of the acids. An excess of Na_2CO_3 should be used in 5.

8. A very good way to neutralize the solution in 5 is to add a slight excess of H_4NHO and warm the solution to expel the ammonia (H_3N), testing the neutrality with litmus paper. The neutralizing is to be performed after the expulsion of CO_2 , as stated in 5.

9. We must seek for HNO_3 and CO_2 in the original solution and not in the solution from 5, as CO_2 has been expelled and HNO_3 has been added.

FIRST PROCESS OF DIVISION INTO GROUPS.

Note.—*Three methods*, selected from many now in use, are given for dividing the acids into groups. *The first* is noted for its simplicity, and is the best for use with a class where one person, either the teacher or one of the pupils, performs the test, and the others note the reactions. *The third* is the one generally selected for individual

research, and is the one that is practically the best for college students to follow in analytical work.

1. To some of the neutral solution previously obtained by 5, add AgNO_3 . The following acids, if present, will be precipitated. We divide them into *two groups*; the *first*, insoluble in HNO_3 ; the *second*, soluble in HNO_3 .

(a) HI , HCl , HCy , HBr , H_2S , H_4FeCy_6 , H_3FeCy_6 , HClO .

(b) H_2CO_3 , H_3PO_4 , HNO_2 , $\text{H}_2\text{S}_2\text{O}_3$, H_2SO_3 , H_2AsO_4 , H_2AsO_3 , $\text{H}_2\text{C}_2\text{O}_4$, $\text{H}_2(\text{C}_4\text{H}_4\text{O}_6)$, $(\text{HO})_4\text{Si}$, $(\text{HO})_3\text{B}$, H_2CrO_4 .

2. To some of the original, neutral solution, add BaCl_2 , the following acids, if present, will be precipitated. As in 1, they are divided into *two groups*; the *first*, soluble in HCl ; the *second*, insoluble in HCl .

(a) H_2CO_3 , HF , H_3PO_4 , $\text{H}_2\text{C}_2\text{O}_4$, H_3AsO_4 , H_3AsO_3 , $\text{H}_2(\text{C}_4\text{H}_4\text{O}_6)$, H_2CrO_4 , $(\text{HO})_4\text{Si}$, $(\text{HO})_3\text{B}$, H_2SO_3 , $\text{H}_2\text{S}_2\text{O}_3$.

(b) H_2SO_4 , H_2SiF_6 .

3. Acids not precipitated by reagents are HClO_3 , HNO_3 , $\text{C}_2\text{H}_4\text{O}_2$.

Note.—Some of the above appear in more than one group.

SECOND PROCESS OF DIVISION INTO GROUPS.

The substance may be a neutral solution, which should be evaporated to dryness, avoiding a high heat; or, it may be a neutral solid.

Place a portion of the substance in an ignition tube with H_2SO_4 , heat gently, and watch for changes.

1. If reactions occur with sudden explosions, there are indications of HClO_3 .

Note.—Owing to a probability of explosions, only a small amount should be used.

2. The occurrence of an odorless, colorless gas, accompanied by brisk effervescence, indicates oxalates or carbonates.

3. The occurrence of a gas possessing odor, but colorless, with some effervescence, indicates—

(a) A sulphide or sulphate, if accompanied by odor of rotten eggs or the fumes as obtained from burning matches.

(b) Odor of almonds or peach blossoms indicates HCy .

(c) Vinegar odors indicate acetates.

(d) Sharp, pungent, irritating odors indicate HF , HNO_3 , or HCl .

4. The occurrence of a gas having a color accompanied by an irritating odor, indicates HNO_2 , HI , or HClO .

5. When these indications are wanting, use the **First Process**.

Third Process of Division into Groups.

BY LA ROY F. GRIFFIN.

1. Add AgNO_3 to the solution containing the free or combined acids, and a small amount of HNO_3 . If a precipitate is formed, shake the solution to make it collect. A precipitate shows the presence of one or more of the following acids: HCl , HCy , HI , HBr .

Filter out the precipitate and treat it with warm H_4NHO , this dissolves AgCl and AgCy , but leaves AgI and AgBr undissolved. To distinguish between HCl and HCy , filter out the precipitate, and neutralize the H_4NHO by HNO_3 . If any AgCl or AgCy were present, a precipitate will reappear. Filter it out, remove it to a piece of porcelain and heat it strongly until it fuses. This decomposes AgCy ,

but leaves AgCl . Now treat the fused mass with a bit of Zn and some dilute H_2SO_4 . The hydrogen set free decomposes the AgCl , and forms HCl . Filter, and test the filtrate for free HCl . (See tests for HCl , p. 67.)

To determine whether HCy is not also present, take some of the original solution, add NaHO to the alkaline reaction, and then a mixture of FeSO_4 , ferrous sulphate, and Fe_2Cl_6 , ferric chloride. This produces a precipitate of mixed ferrous hydrate, Fe_2HO , and ferric hydrate, Fe_2FeHO . Add HCl to this, and the precipitate dissolves. A blue precipitate will appear almost immediately if HCy was present, otherwise the solution assumes the color of the chlorides of Fe .

The previous steps have shown whether HI and HBr are present, or absent. Confirm their presence by the special tests for these acids applied to portions of the original solution, particularly Tests 2 and 4. (See p. 68.)

NOTE.—There is no one test that will determine the presence of Br when I is also present. It can be determined with satisfaction only from comparison of all the tests, observing the colors with great care.

2. To a portion of the original solution, add barium chloride, BaCl_2 . The following acids are precipitated as the corresponding salts: H_2SO_3 , H_2SO_4 , H_2CO_3 , $\text{H}_3\text{C}_2\text{O}_4$, H_3PO_4 , H_2CrO_4 , H_3BO_3 , $\text{H}_6\text{C}_4\text{O}_8$, $\text{H}_8\text{C}_8\text{O}_7$.

The sulphites and carbonates make BaSO_3 and BaCO_3 , with BaCl_2 , but they can be best tested under 4, since they effervesce. (See 4.)

A fine white precipitate, which filtered out can not be dissolved in warm dilute HCl , indicates H_2SO_4 . (See p. 66.)

If H_2SO_4 has been found to be present, it must be removed before proceeding to test for oxalic acid, $\text{C}_2\text{H}_2\text{O}_4$. It is done by adding HCl to acid reaction, and then just sufficient BaCl_2 to precipitate the H_2SO_4 as BaSO_4 , *carefully avoiding much excess*. The BaSO_4 is then filtered out, leaving the $\text{H}_2\text{C}_2\text{O}_4$ in the filtrate.

Add H_4NHO to the alkaline reaction. If any precipitate appears, filter it out, as it will obscure the test for $\text{H}_2\text{C}_2\text{O}_4$. The special tests for $\text{H}_2\text{C}_2\text{O}_4$ may now be applied, particularly the first. (See p. 70.)

To determine the presence of H_3PO_4 , the original solution may be tested according to the special tests for the acid. (See p. 69.)

H_3PO_4 may also be tested as follows: If the original solution is not acid, add acetic acid to acid reaction, and a few drops of Fe_2Cl_6 . A yellowish white precipitate of ferric phosphate, $\text{Fe}_2(\text{PO}_4)_3$, shows the H_3PO_4 .

When the solution is acid, add H_4NHO until it is alkaline; then add HCl drop by drop until all the precipitate that formed disappears; then add ammoniac acetate. As before, ferric chloride will now test the solution.

Nitric acid solution of molybdate of ammonia, $(\text{H}_4\text{N})_2\text{MoO}_4$, is also a good test for H_3PO_4 .

The test for chromic acid, H_2CrO_4 , need not be applied unless the solution is colored. If colored, add acetic acid and apply the special tests, particularly the second. (See p. 69.)

To test for H_3BO_3 , dissolve some of the salt in a small amount of H_2SO_4 , and then apply the special tests. (See p. 69.)

NOTE.—Tartaric and citric acid are best tested for under organic acids. (Sec 6.)

3. The Deflagrating Acids. NOTE 1.—Deflagration is a rapid combustion from the union of charcoal with a substance melted upon it.

NOTE 2.—If the substance is a solution and acid, apply the special test for HNO_3 at once.

Heat a portion of the dry salt upon charcoal before the blowpipe. Deflagration shows the presence of a nitrate or a chlorate. If the substance is a solution, a small quantity must be evaporated to dryness, to apply this test.

When deflagration ensues, test for HNO_3 . (See p. 66.)

If a salt tested proves not to be a nitrate, it may be tested for HClO_3 , or if there is any suspicion that more than a single acid is present. (See special test, p. 67.)

4. Effervescing Acids. These acids make salts which, upon decomposition by a stronger acid, give off a gas.

Add to some of the original substance in solution a little water, then add a few drops of HCl. Effervescence indicates the presence of one of the following acids: H_2CO_3 , H_2S , H_2SO_3 , HCy.

NOTE 1.—Usually, when HCl is added to a sulphide, the salt is decomposed with the precipitation of S. A white milky precipitate therefore shows a sulphide.

NOTE 2.—When HCl is added to the solution of a sulphite, sulphurous acid fumes are evolved without any deposition of S; but when added to a hyposulphite, sulphurous acid is given off and a milky pp. of S occurs.

NOTE 3.—A silicate similarly treated often produces a thick gelatinous precipitate of silicic acid, H_4SiO_4 .

Next, place a small portion of the substance to be tested in a test tube and add dilute HNO_3 , a colorless gas given off shows H_2CO_3 . Test for that acid. (See p. 67.)

When the presence of a sulphide has been indicated by the preliminary test, add dilute H_2SO_4 to some of the original substance, and test the gas liberated with paper moistened with solution of lead acetate. (See tests for H_2S , p. 70.)

Sulphites are usually recognized in the preliminary tests by the odor. In doubtful cases, add dilute H_2SO_4 to the original substance, and the odor of SO_2 will show its presence. (See tests for H_2SO_3 , p. 66.)

5. Mineral Acids. H_2SiO_3 and HF usually appear in insoluble salts; a few are soluble.

The insoluble salts are treated by fusing a small portion combined with about four parts of mixed K_2SO_3 and Na_2CO_3 . This changes the salt to a soluble form, so that it can be dissolved in water.

Add to the solution HCl and boil. A gelatinous precipitate shows H_4SiO_4 . (See p. 69.)

When H_4SiO_4 is not found, test the original substance for HF by etching. (See p. 68.)

6. Organic Acids. These acids are all compounds of carbon. Heat a small portion upon a piece of porcelain. Charring indicates an organic acid.

NOTE.—Be careful to distinguish between blackening, such as appears when certain metallic salts are heated, and charring.

Moisten a small portion of the solid substance with alcohol and H_2SO_4 . Acetic ether is given off when an acetate is present, and it may be recognized by its odor. (See tests, p. 71.)

Heat a small portion carefully upon porcelain. If the odor of tartaric acid is perceived, apply the special tests. (See p. 71.)

Test some of the original solution for citric acid. (See p. 71.)

Tannic and gallic acids are recognized by adding some ferric salt, as Fe_2Cl_6 , to the solution. Black ink is produced if either is present.

Add to the solution found to contain one of these acids, a small amount of HCl , and then a solution of gelatine. A curdy white precipitate shows tannic acid; the absence of a precipitate shows gallic acid.

If neither of the above acids have been found, test for oxalic acid. (See p. 70.)

CHAPTER X.

SPECIAL TESTS FOR DIFFERENT ACIDS.

Note.—Remember, when a substance has been added to a solution, the character of the solution is changed, and you must search in the original solution to ascertain whether or not the kind of substance just added was originally there.

Sulphuric (H_2SO_4).

Add BaCl_2 , white pp. (BaSO_4), insoluble in HCl or other acids.

Thiosulphuric ($\text{H}_2\text{S}_2\text{O}_3$).

1. Add HCl , pp. of Sulphur, and sulphurous acid fumes.

2. Add BaCl_2 , white pp., soluble in HCl , leaving a residue of S.

Sulphurous (H_2SO_3).

Add BaCl_2 , white pp. (BaSO_3), soluble in HCl ; when HNO_3 is added to pp. it forms BaSO_4 , insoluble in acids.

Note.—The characteristic odor is, in many cases, a sufficient test for this acid.

Nitric (HNO_3).

1. Add indigo solution and H_2SO_4 , and apply heat; the blue of the solution will fade.

2. Add solution of FeSO_4 , then carefully add H_2SO_4 ; brown ring or spot appears where the H_2SO_4 meets the solution.

Note.—Form a solution of FeSO_4 , when wanted, by adding a clear crystal of the salt to H_2O and applying heat.

Nitrous (HNO_2).

1. Acidify the solution with acetic acid and add solution of potassium permanganate; the solution is decolorized.

2. Acidify as in 1, and add AgNO_3 , white pp. (AgNO_2), soluble in acids and excess of H_2O .

3. Colors starch paste, to which KI has been added, a deep blue.

4. Nitrites are decomposed by acids, with evolution of red fumes.

Hydrochloric (HCl).

1. Add AgNO_3 , white pp. (AgCl), insoluble in HNO_3 , soluble in H_4NHO , turns violet in sunlight. (Usually boil the solution in HNO_3 .)

2. Add H_2SO_4 and MnO_2 , and heat. Cl_2 fumes appear, which bleach moist litmus paper.

3. Add $\text{Hg}_2(\text{NO}_3)_2$, *mercurous nitrate*, white pp. (HgCl); pp. is insoluble in HNO_2 , but is blackened by H_4NHO .

Chloric (HClO_3).

Add strong H_2SO_4 , yellowish gas (Cl_2O_4) is liberated with explosive violence. Use a small quantity and without applying heat.

Note.—Deflagration is always an indication of nitrates or chlorates.

Carbonic (H_2CO_3) and the Carbonates.

1. Add HCl or HNO_3 , effervescence; tested by passing gas through lime water (Ca_2HO), white pp. (CaCO_3).

2. Gas set free in 1 is odorless, and will extinguish a lighted match.

Hydrobromic (HBr).

1. Add AgNO_3 , yellowish-white pp. (AgBr), insoluble in HNO_3 ; slightly soluble in H_4NHO , easily soluble in KCy .

2. Add H_2SO_4 and MnO_2 , and heat; brown vapors of Br_2 appear.

3. Add chlorine water, then a few drops of CS_2 ; shake, and CS_2 will be colored brownish-yellow. Avoid excess of chlorine water.

4. If solution of starch is brought into contact with Br_2 , yellow bromide of starch is formed.

Hydriodic (HI).

1. Same as 1 for HBr , yellow pp. (AgI).

2. Same as 2 for HBr , violet fumes of I_2 .

3. Same as 3 for HBr , CS_2 colored violet.

4. Same as 4 for HBr , blue iodide of starch.

5. Mercuric chloride, HgCl_2 , gives a salmon-colored pp., rapidly becoming scarlet, HgI_2 .

HgI_2 heated upon a filter turns yellow.—*G*.

Hydrocyanic (HCy or HCN).

1. Add a few drops of KHO and FeSO_4 ; then add HCl , forms Prussian blue.

2. Has the odor of bruised peach leaves.

3. In 1, if the solution is weak, the liquid assumes a green color and deposits the blue after a time.

Hydrofluoric (HF).

1. Irritating odor upon adding H_2SO_4 and heating.

2. The "etching process" is a test.

Phosphoric (H_3PO_4).

1. Add AgNO_3 , light-yellow pp. (Ag_3PO_4), soluble in H_4NHO , HNO_3 , and in acetic acid.
2. Add MgSO_4 with H_4NCl and H_4NHO , white pp. (MgH_4NPO_4), soluble in acids.

Chromic (H_2CrO_4).

1. Add H_2S , green pp. (See 4).
2. Add lead acetate, $\text{Pb}_2\text{C}_2\text{H}_3\text{O}_2$, yellow pp. (PbCrO_4).
3. Add AgNO_3 , brownish-red pp. (Ag_2CrO_4).
4. Add $(\text{H}_4\text{N})\text{HS}$, green pp. (Cr_26HO).

Boric (H_2BO_3).

1. Add solution to alcohol, flame tinged green. (See **Flame Tests**, p. 24.)
2. Mixture of glycerine and boric acid brought into the flame shows green tinge.
3. Acid borates form a white pp. (Ag_3BO_3), with AgNO_3 . Neutral borates form a brown oxide of silver.

Hypochlorous (HClO).

1. Add AgNO_3 , pp. (AgCl).
2. Acidulate with acetic or HCl acids, and Cl_2 gas will be evolved, showing bleaching effects.

Arsenic (H_3AsO_4) and Arsenious (H_3AsO_3)

Are to be tested the same as for Arsenic (As), which see.

Note.—Remember that the sulphides of CdS , SnS_2 , and Sb_2S_3 are insoluble in $(\text{H}_4\text{N})_2\text{CO}_3$, while As_2O_3 is soluble.

Silicic (H_4SiO_4).

1. Fuse the solid silicate on charcoal with Na_2CO_3 ; dissolve in HCl , and evaporate to dryness. If a white

powder, SiO_2 , insoluble in HCl and soluble in KHO , be found, some form of silicic acid is present. Generally, the solution and pp. are boiled in KHO .

2. When the acid is in solution, add HCl to a strong solution of an alkaline silicate; the silicate sets in a jelly-like mass. When solution is weak, add HCl and evaporate to dryness. You obtain a white powder, same as in 1.

Hydrosulphuric (H_2S).

1. To a few drops of HCl , add some of the H_2S solution. H_2S will be evolved, well known by its smell.

2. All compounds containing sulphur, when heated with Na_2CO_3 on charcoal, yield Na_2S , which, when moistened, stains silver brown or black.

H_2S in the gaseous state quickly blackens papers moistened with a solution of lead acetate.—*G*.

Hydroferrocyanic (H_4FeCy_6) and Hydroferricyanic (H_3FeCy_6)

Are tested by the tests for iron. See Fe, 3, *ferrous salts*, and 1, *ferric salts* (pp. 54, 55).

Oxalic ($\text{H}_2\text{C}_2\text{O}_4$) or (H_2Ox).

1. Add CaCl_2 , white pp. (CaC_2O_4), calcium oxalate, soluble in HCl , insoluble in acetic acid.

2. Effervesces with H_2SO_4 , colorless and odorless gas.

3. Add AgNO_3 , white pp. ($\text{Ag}_2\text{C}_2\text{O}_4$), oxalate of silver.

Tartaric ($\text{H}_2(\text{C}_4\text{H}_4\text{O}_6)$ or (H_2Tr).

Note.— Make a normal solution of the acid as follows: 1 part acid, 8 parts water, 2 parts alcohol. Neutralize the solution by adding solution Na_2CO_3 , then proceed as in 1 or 2.

1. Add AgNO_3 , white pp. ($\text{Ag}_2\text{C}_4\text{H}_4\text{O}_6$), tartrate of silver, turns black on boiling.

2. Add CaCl_2 , white pp. ($\text{CaC}_4\text{H}_4\text{O}_6$), tartrate of calcium, soluble in cold solution of KHO . Heat, and the pp. returns.

Citric ($\text{H}_3(\text{C}_6\text{H}_5\text{O}_7)$).

1. See note above. Test same as No. 1 for Tartaric. Solution does not blacken when boiled.

2. Add lead acetate, $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$, white pp.

3. Add CaCl_2 and boil, white pp. ($\text{Ca}_3(\text{C}_6\text{H}_5\text{O}_7)_2$), not soluble in KHO .

Acetic ($\text{H}(\text{C}_2\text{H}_3\text{O}_2)$).

1. To a solution made neutral by adding an acid or an alkali, add a neutral solution of Fe_2Cl_6 , a red solution is formed; not decolorized by HgCl_2 , but decolorized by HCl .

2. Add HCl and then heat: odor of vinegar is evolved.

3. Add alcohol and a small amount of H_2SO_4 , the odor of acetic ether is produced.

CHAPTER XI.

TREATMENT OF EXCEPTIONAL SALTS.

BY LA ROY F. GRIFFIN.

The oxalates, borates, and phosphates of barium, strontium, calcium, and magnesium often behave in such a manner upon the addition of the Group Reagent as to mislead the analyst. A precipitate is produced by H_4NHO , instead of the regular Group Reagent. A special search must therefore be made for the metals.

The following indications are those requiring such special tests:

(a) When the original substance, insoluble in H_2O , is soluble in HCl , and is precipitated by H_4NHO .

(b) When H_4NHO produces a white or gray precipitate, and the metals of the ammonia group are absent.

(c) When *preliminary tests* indicated some one of these metals, but *ordinary tests* fail to confirm the indications.

(d) When the tests for acids show the presence of one of these, and the salts have not already been proved absent.

If any, or all, of the preceding indications are present, pursue the following process:

Carefully test for the presence of oxalic acid (see p. 70). If it is found, ignite a portion of the original substance upon porcelain—this changes the salt to a carbonate. Dissolve the carbonate in HCl , thus making a chloride, and then pursue the usual process of testing for the metal.

Carefully test for phosphoric acid, H_3PO_4 , and, if present, dissolve a portion of the original substance in HCl , neutralize with ammoniac acetate ($\text{H}_4\text{NC}_2\text{H}_3\text{O}_2$), then add Fe_2Cl_6 until all the phosphate has been changed to ferric phosphate ($\text{Fe}_2(\text{PO}_4)_2$). The metal will remain as a chloride, when test for the metal as usual.

Carefully test for boric acid in the usual way. If found, dissolve some of the original substance in HCl, and add H_2SO_4 and alcohol. This precipitates the metal as a sulphate. Filter and treat the precipitate as follows:

Boil the precipitate in a mixture of Na_2CO_3 and K_2CO_3 for about five minutes; this turns CaSO_4 and SrSO_4 to CaCO_3 and SrCO_3 ; BaSO_4 remains unaffected. Filter out the precipitate of SrCO_3 , CaCO_3 , and BaSO_4 , and wash it with hot water; then treat it with dilute HCl to dissolve the Sr and Ca. Now test the undissolved portions for Ba (see p. 56), and the solution for Sr and Ca (see pp. 56, 57).

Magnesium borate demands a special treatment, because MgSO_4 is soluble in alcohol. Upon adding H_2SO_4 and filtering out the BaSO_4 , SrSO_4 , and CaSO_4 , the MgSO_4 will pass through into the filtrate. Mg may then be precipitated as MgO_2H_2 by adding NaHO. Dissolve the MgO_2H_2 in HCl, add H_4NCl and H_4NHO . If the solution is not yet clear, filter and test the clear liquid for Mg. (See p. 57.)

STUDENTS' TABLE—CHEMICAL APPARATUS.

An excellent table for Analytical work can be arranged as shown in the cut opposite the title page. The table is provided with drawers upon either side. The top of the table is divided into sections for individual work, by wide brackets that support shelves above. Only one shelf is shown in the engraving; it is often very convenient to have two shelves, placed one above the other. Each student should be supplied with lamp, ring-stand, test-tube rack, test-tube holder, reagents, test-tubes, evaporating dishes, flasks, retorts, funnels, glass and rubber tubing, swabs, pipettes, and such utensils as are often needed. It is not necessary to duplicate cork-borers, mortars, hydrometers, balances, Woulff bottles, etc.

Some pieces of chemical apparatus are shown on page 86. The upper part of the cut, from the left, represents a reagent bottle, flask, test-tubes, pipettes, hydrometer jar, graded centimeter jar, mortar, thistle-tubes, and hydrometer. Below these we see graduate with lip, nest of beaker glasses, Woulff bottle, oxygen retort, tabulated glass retort, balances, test-tube holder, ring-stand, lamp, blow-pipe, evaporating dish, and a set of cork-borers. Every laboratory should possess an "Alcohol Blast-Lamp" for glass bending and heating.

The publishers of this book have arranged with a reliable firm to supply pure chemicals and perfect apparatus to such as desire them. Orders for chemicals and apparatus, or inquiries concerning the same, addressed to them, will receive prompt and careful attention.

APPENDIX.

METRIC MEASUREMENTS.

1 mm.	=	.0394 in.	1 g.	=	15.43235 grains.
1 cm.	=	.3937 in.	1 grain	=	.0648 g.
1 in.	=	2.539954 cm.	1 lb. av.	=	453.59 g.
1 cu. in.	=	16.386176 cc.	1 oz. av.	=	28.34954 g.
1 cc.	=	.06103 cu. in.	1 gal. U. S.	=	231 cu. in.
1 ^l	=	61.02705 cu. in.	1 gal. Imp.	=	277 $\frac{1}{4}$ cu. in.

SOLUTION OF AMMONIO-SULPHATE OF COPPER.

To a solution of pure sulphate of copper, 1 part to 20 of water, add ammonia until the blue precipitate, at first formed, is nearly but not quite re-dissolved. Filter, and preserve the liquid as an arsenicum reagent, labeling it carefully as above. Treat solution of nitrate of silver, 1 part to 40 of water, in the same way, and label it "*Solution of Ammonio-nitrate of Silver.*"—*Attfield.*

TEST FOR LEAD IN TIN.

A person can satisfactorily determine whether tin contains lead in sensible quantity, by the following process: Place a drop of strong nitric acid on the tin surface, by means of a glass rod, or even a wooden toothpick, and rub the acid over an area equal to the size of a penny. Warm it very gently until it is dry, and then place two drops of a solution of iodide of potassium on this spot. If the tin contains lead, the bright yellow iodide of lead will form. The test can be easily and rapidly made, and the result is very decisive.

The above test should be made with all tin used for canning purposes. Discard any specimen that shows the presence of lead, as fatal results are liable to follow its use for meats or vegetables. Roofing tin contains lead.

MARSH'S TEST FOR ARSENIC.

Arrange the evolution flask the same as in the generation of hydrogen, *taking especial care that every piece of apparatus is clean and that the chemicals used are pure.* Except in rough experiments, dry the escaping gas by inserting a larger piece of glass tubing in the delivery tube, such piece to contain fragments of calcium chloride, CaCl_2 , or pieces of caustic potash, KHO . Sometimes a little cotton wool is placed in the end of the drying tube, next the flask. This helps to stop any liquid that may be carried over mechanically.

After the hydrogen gas has been flowing a few minutes, or long enough to expel *all* of the air from the flask, light the hydrogen at end of delivery tube. (The end of the delivery tube should be previously drawn to a narrow aperture by using a flame.) Test the purity of your materials by holding a piece of glazed porcelain in the burning hydrogen jet. If no spot is formed, your materials are presumably pure or free from arsenic.

The solution under examination for arsenic is then gradually added to the contents of the flask, by means of the funnel tube. If arsenic is present, it will appear in the flame as arseniuretted hydrogen, giving to the flame a pale lavender tint, and pieces of white porcelain, placed in the flame, become covered with a brown coating of metallic arsenic. Several of these spots should be collected for further tests.

Note 1.—Remember that arseniuretted hydrogen gas is *very poisonous*, and will produce *fatal results* if much of it is inhaled. Perform the experiment under the "gas duct."

Note 2.—It is better to generate the hydrogen at a low temperature. When the liquid becomes too hot, insoluble sulphide of arsenic is formed. Also avoid nitrates, nitrites, chlorides, and free chlorine.

Another way to produce the arsenical spot is to have a delivery tube of hard glass, about two feet long. By strongly heating the hard glass tube near the middle, in the gas flame, the arseniuretted hydrogen will be decomposed and the arsenic will be deposited farther along in the tube, in a dark, mirror-like form. The escaping gas may be passed through a dilute solution of silver nitrate, AgNO_3 . The escaping arseniuretted hydrogen is decomposed, silver is precipitated, and the arsenious acid, $\text{As}(\text{HO})_3$, is retained in the solution.

Note 1.—A solution of arsenic in milk, or some similar material, may give forth so much CO_2 as to interfere with the proper evolution of the hydrogen. In such cases, neutralize the solution by adding pure H_2SO_4 .

Note 2.—Antimony and arsenic are very similar in their reactions and in their appearance as a metallic spot; consequently, tests upon the metallic spot must be made to determine its nature.

Testing the Spot for Arsenic.

1. Add a drop of $(\text{H}_4\text{N})_2\text{S}$, spot turns yellow.
2. Add HCl , spot does not dissolve.
3. Add hot HNO_3 , spot dissolves clear. To this, add AgNO_3 , no change in color. Treat the solution by blowing vapor of H_3N from the mouth of the ammonia bottle upon the spot, the solution turns brick-red.
4. Add a solution of chloride of lime (CaCl_2O_2 , CaCl_2) to the spot, the spot dissolves.

Note.—An antimony spot will not dissolve.

ORGANIC IMPURITIES IN WATER.

Make a solution as follows:

- 12 grains caustic potash, in stick form.
- 3 grains permanganate of potash.
- 1 ounce pure water.

Put one drop in a small quantity of H_2O to be tested. If the test is decolorized, it indicates organic impurities.

If not decolorized at once, set aside in a moderately warm place, for a little time. If not decolorized then, organic impurities are undoubtedly absent.

Drinking water often needs to be tested for the presence of Cl_2 , by AgNO_3 , which produces a white precipitate of AgCl if Cl_2 is present. For nitrites, by stirring in a small quantity of starch paste containing KI ; the nitrites decompose the KI , and the free I_2 produced blues the water by forming a compound with the starch; and for free H_3N , by Nessler's reagent. Either organic impurities or one of the above substances give rise to a suspicion of sewage contamination.—G.

An additional test for lead in water is to fill a glass tumbler with the water to be tested, and set it upon a sheet of white paper. Add a drop or two of dilute HNO_3 and some H_2S . A dark brown tinge, seen by looking through from above, will indicate the presence of lead.

Nessler's Test for Ammonia in Water.

BY LA ROY F. GRIFFIN.

Prepare the reagent as follows (the quantities are given for one litre; a smaller amount will require the same proportion): Dissolve 34 grms. KI in 100 cc. of water, and add a saturated solution of HgCl_2 until the precipitate first formed ceases to dissolve. Filter. Dissolve 160 grms. KHO in just sufficient water to dissolve it, and add the solution to the filtrate. Now dilute the whole to the volume of one litre and add 5 cc. of saturated solution of HgCl_2 . Let the solution stand so that any precipitate may settle, and then decant the clear liquid for use. The addition of HgCl_2 is to "sensitize" the solution. The sensitiveness is lost by standing; consequently, the HgCl_2 should be added when the solution is wanted for use.

NOTE.—This reagent can be used for quantitative tests by standardizing, that is, determining the amount of the reagent required to neutralize a given quantity of ammonia.

To test for free ammonia, add a small amount of the reagent to the clear solution; a brown precipitate is formed.

When some salt of ammonia is present, place the substance to be tested in a clean flask, add a small amount of water and a piece of solid NaHO. Fit the flask with a rubber stopper and a bent delivery tube, place the end of the delivery tube in a test tube containing some of the reagent, and heat the flask to boiling. If the substance treated was a salt of ammonia, the precipitate will appear.

NOTE.—Certain substances containing N and H uncombined, form H_3N , when distilled. These require separate examination.

CHEMISTRY WITHOUT APPARATUS.

When one is engaged in qualitative chemical analysis, it is necessary to change vessels at almost every reaction, or else be compelled to resort to frequent washing, which, if it be not properly performed, may spoil the results of the subsequent reaction. One of the best means that has been tried of getting over this difficulty consists in the use of the smoked capsules proposed by Mr. Violette. By smoked capsule is meant a small porcelain saucer covered with a layer of lampblack by putting it into the flame of a candle. A drop of water or of a saline solution, carefully deposited in this capsule, assumes therein the form of a globule which is as limpid as crystal, and which does not adhere to the lampblack. The addition to this globule of another drop of saline solution or a particle of a solid reagent produces therein all the phenomena of coloration, precipita-

tion, and crystallization with perfect clearness. The eye is capable of following in it the least changes (which are rendered still more manifest through the lenticular magnification) without having to look through the glass sides of what are usually used as receptacles.

After the phenomena has been observed, the globule is thrown out through a slight blow on the capsule. The latter will be found clean, without residuum, and perfectly fitted for the examination of another reaction without any mixture with the preceding. The vessel is, so to speak, clean without the necessity of cleaning it, and there need be no fear of any of those contaminations, even slight ones, that sometimes spoil analytical results in the ordinary vessels used.

The capsule, which is only three-quarters of an inch in diameter, is nothing else than one of those small porcelain saucers used for water colors. In order to put it into the flame, it must be grasped with pincers; but the operation may be more easily performed by gluing a thin cork disk to it, and sticking a pin into this for a handle. For smoking one of these capsules properly it is necessary to use precaution. It should be thrust into the upper third of the flame of a candle several times, and be allowed to cool in the intervals. It is necessary to wait until the capsule is cold before depositing the globule in it, for otherwise it would get wet. The carbonaceous coating is at once made wet by acid, alcoholic, and ethereal liquids, and it is only aqueous solutions that assume a globule form upon it.

This mode of operating may be still further simplified by taking advantage of the property that the leaves of some plants possess of not being wet by water and aqueous saline solutions. Among such leaves, those of the nasturtium (*Tropæolum*) have a form that especially adapts them to this use. When one of these is held by its petiole, its upper surface exhibits a depression in which one can easily deposit a globule, and proceed exactly as with the smoked capsule. When the leaf becomes wet, after a few reactions, nothing is easier than to substitute another one for it.—*La Nature*.

ANTIDOTES.

Alkalies. Vinegar or lemon juice would be a good antidote for a caustic alkali.

Antimony. There should be administered an infusion, either of tea, nutgalls, oak bark, or some astringent which forms the insoluble tannate of antimony. The stomach pump should be used as soon as possible.

Arsenic. Freshly prepared ferric hydroxide ($\text{Fe}_2\text{O}_3\cdot\text{H}_2\text{O}$), made by adding H_4NHO to a solution of Fe_2Cl_6 . Filter, and wash the precipitate. The latter is then ready for use.

MgO , magnesia, is an antidote. In a few moments, follow the magnesia with a teaspoonful of mustard in a cup of warm water. Soon after giving the mustard, administer a dose of castor oil.

The stomach pump should be used.

Barium. Any sulphates, such as those of magnesium and sodium, would be good.

Carbolic Acid. The best antidote is castor oil or olive oil freely administered.

Copper. Raw eggs should be swallowed, as the albumen forms a compound insoluble in water. Use mustard for emetic, and apply the pump.

Cyanides. These poisons act very quickly. Emetics and the stomach pump should be used at once. Apply a stream of cold water to the spine.

Lead. When poisoned by a soluble sulphate of lead, use Epsom salts, sulphate of sodium, or alum. These produce vomiting, which should be followed by the application of the stomach pump.

Mercury. Albumen gives a white pp.; therefore, when poisoned by corrosive sublimate, administer the white of eggs while waiting for a pump.

Nitric Acid. Use a solution of carbonate of sodium, washing soda, or a mixture of magnesia and water, administering the substance slowly enough to allow the escape of the CO_2 set free.

Oxalic Acid. Use chalk and water, emetics and the pump being used as soon as possible.

Silver. Solution of common salt should be administered when large doses of AgNO_3 have been swallowed; also, produce vomiting by the use of an emetic.

Sulphuric Acid. See **Nitric Acid.**

Tin. Use white of eggs; induce vomiting, and apply the pump.

Zinc. Use solution of carbonate of sodium, followed by the white of an egg.

TABLE OF REAGENTS.

Showing where pp. are formed, and their color, as recorded in the previous pages of this volume.

d = dark.	y = yellow.	or = orange.	fl = flesh colored.
w = white.	g = green.	pur = purple.	l = light.
b = black.	br = brown.	bl = blue.	r = red.

A combination of the letters is used to indicate "middle colors."

The reagent is given at the left, followed by the material tested, and the color of pp.

H₂SO₄. Pb-w. Ba-w.

(H₄N)₂CO₃. Pb-w. Ba-w. Sr-w. Fe-w. Fe-br. Ca-w.

Mg-w.

K₂Cr₂O₇. Pb-y. Bi-y. Ba-y.

K₂CrO₄. Ag-d.r. Hg-or. Ba-y.

KI. Pb-y. Ag-y.w. Hg-y.g. Hg-y.

HCl. Ag-w. Hg-w. Pb-w. As-y.

H₂S. Ag-b. Hg-b. Sb-or. Sr-y. Bi-b. Cu-b. Cr-g.
Au-br. H₂CrO₄-g. Pb-b. Hg-w. As-y. Sn-br. Cd-y.

KHO. Ag-br. Ni-g. Mn-w. Sn-w. Bi-w. Cu-g.

KCy. Ag-w. Cu-g.y. Cd-w. Ni-br.

NaHO. Pb-w. Hg-b. Hg-y.

(H₄N)₂S. As-y. Hg-b. Cr-bl.g. Al-w.

SnCl₂. Hg-grey. Hg-w. Au-pur.

(H₄N)HO. Bi-w. Cu-g.bl. Fe-br. Ni-g. Co-bl. Mn-w.
Zn-w. Hg-w. Sb-w. Sn-w. Cd-w. Ni-g. Zn-w.

HgCl₂. Sn-w.

Na₂CO₃. Sr-w.

H₄NOx. Sr-w. Ca-w.

(H₄N)HS. Cu-d. Fe-b. Ni-b. Mn-fl. Zn-w. Co-b.

H₂CrO₄-g.

K₄FeCy₆. Fe-b. Cu-r.br. Fe-w.

Lead Acetate. Cr-y. H₂CrO₄-y. Citric-w.

K₂Fdcy. Fe-bl.

AgNO₃. Cr-br. HCl-w. HBr-y.w. HI-y. H₃PO₄-y.
H₂CrO₄-br. Tartaric-w. Citric-w. Oxalic-w. As-y. As-r.b.

Na₂PHO₄. Mg-w. Ag-y. Al-w. Ba-w.

Hg₂(NO₃)₂. HCl-w.

Tartaric Acid. K-w.

Ammonio-Sulphate of Copper. As-g. As-l.b.

Ammonio-Nitrate of Silver. As-y. As-r.

PtCl₄. K-y. H₄N-y.

H₂SiF₆. Na-w.

BaCl₂. H₂SO₄-w.

FeSO₄. HNO₃-br.

CaCl₂. Tartaric-w. Citric-w. Oxalic-w.

Fe₂Cl₆. Acetic-r.

TABLE INDICATING BASES.

P stands for precipitate. N for no precipitate.

REAGENTS USED.			METALS INDICATED.
Na ₂ CO ₃ .	H ₃ N.	KHO.	
N.	N.	—	K, Na, H ₄ N.
P.	N.	—	Ba, Sr, Ca.
P.	P.	Blue P.	Cu, Co.
P.	P.	Green P.	Fe, Ni.
P.	P.	Brown P.	Fe, Ag.
P.	P.	Black P.	Hg, Au.
P.	P.	Yellow P.	Hg, Au, Pt.
P.	P.	White P.	Pb, Zn, Sn, Al, Sb. (The precipitate dissolves.)
P or N.	P.	White P.	Mg, Bi, Fe, Cd, Mn. (The precipitate does not dissolve.)

Modified from *Rains*.

TABLE OF ELEMENTS.

The atomic weight is according to Roscoe and Schorlemmer.
In rare cases higher terms of valence are found.

NAME.	SYMBOLS AND VALENCE.	ATOMIC WEIGHT.
Aluminum	Al 4	27.3
Antimony	Sb 3, 5	122
Arsenic	As 3, 5	74.9
Barium	Ba 2	136.8
Bismuth	Bi 3, 5	210
Boron	B 3,	11
Bromine	Br 1, 3, 5	79.75
Cadmium	Cd 2	111.6
Calcium	Ca 2	39.9
Carbon	C 2, 4	11.97
Chlorine	Cl 1, 3, 5	35.37
Chromium	Cr 4, 6	52.4
Cobalt	Co 2, 4	58.6
Copper	Cu 2	63
Fluorine	F 1	19.1
Gold	Au 1, 3	196.2
Hydrogen	H 1	1
Iodine	I 1, 3, 5	126.53
Iron	Fe 2, 4, 6	55.9
Lead	Pb 2, 4	206.4
Magnesium	Mg 2, 4, 6	23.94
Manganese	Mn 2	54.8
Mercury	Hg 2	199.8
Nickel	Ni 2, 4	58.6
Nitrogen	N 1, 3, 5	14.01
Oxygen	O 2	15.96
Phosphorus	P 1, 3, 5	30.96
Platinum	Pt 2, 4	196.7
Potassium	K 1	39.04
Silicon	Si 4	28
Silver	Ag 1	107.66
Sodium	Na 1	22.99
Strontium	Sr 2	87.2
Sulphur	S 2, 4, 6	31.98
Tin	Sn 2, 4	117.8
Zinc	Zn 2	64.9

TABLE SHOWING THE SOLUBILITY IN

W=soluble in water.

A = sparingly soluble in water,

S = sparingly soluble in water.

increased by acids.

B = insoluble in water — soluble
in acids.

No.	Salts.	Acid.	Base.								
			K ₂ O	Na ₂ O	(H ₄ N) ₂ O	BaO	SrO	CaO	MgO	Al ₂ O ₃	MnO
1	Hydrate	H(HO)	W	W	W	W	S	S	B	B	B
2	Sulphide	H ₂ S	W	W	W	W	W	W	A	..	B
3	Chloride	HCl	W	W	W	W	W	W	W	W	W
4	Iodide	HI	W	W	W	W	W	W	W	W	W
5	Bromide	HBr	W	W	W	W	W	W	W	W	W
6	Cyanide	HCy	W	W	W	S	W	W	W	..	B
7	Ferrocyanide	H ₄ Fcy	W	W	W	S	W	W	W	B	B
8	Ferricyanide	H ₃ Fdcy	W	W	W	W	..	W	W	..	O
9	Fluoride	HF	W	W	W	B	B	B	B	B	B
10	Silicate	H ₂ SiO ₃	W	W		E	E	E	O	B	B
11	Fluo-Silicate	H ₂ SiF ₆	S	W	..	O	W	A	W	..	W
12	Sulphate	H ₂ SO ₄	W	W	W	O	O	SO	W	W	W
13	Sulphite	H ₂ SO ₃	W	W	W	B	A	A	A	W	S
14	Nitrate	HNO ₃	W	W	W	W	W	W	W	W	W
15	Chlorate	HClO ₃	W	W	W	W	W	W	W	W	W
16	Phosphate	H ₃ PO ₄	W	W	W	B	B	B	B	B	B
17	Arsenite	H ₃ AsO ₃	W	W	W	S	S	B	B	..	B
18	Arsenate	H ₃ AsO ₄	W	W	W	B	B	B	B	B	B
19	Chromate	H ₂ CrO ₄	W	W	W	B	S	S	W	..	W
20	Acetate	C ₂ H ₄ O ₂	W	W	W	W	W	W	W	W	W
21	Tartrate	C ₄ H ₆ O ₆	W	W	W	B	B	B	S	W	A
22	Oxalate	C ₂ H ₂ O ₄	W	W	W	B	B	B	S	B	S
23	Borate	H ₂ BO ₃	W	W	W	B	B	B	S	B	B
24	Carbonate	H ₂ CO ₃	W	W	W	B	B	B	B	..	B

WATER AND ACIDS OF THE COMMON SALTS.

P = soluble in small amount of water—precipitated in large amount of water, prevented by presence of free acid. O = insoluble in water and acids.
 E = soluble in part, not completely, by acids.

FeO	Fe ₂ O ₃	CuO	CoO	NiO	ZnO	CdO	PbO	Ag ₂ O	Hg ₂ O	HgO	SnO	SnO ₂	Bi ₂ O ₃	PtO ₂	Sb ₂ O ₃	Cr ₂ O ₃	No.
B	B	B	B	B	B	B	B	B	B	B	B	E	B	B	B	E	1
B	..	B	B	B	B	B	B	B	B	B	B	B	B	B	B	..	2
W	W	W	W	W	W	W	S	O	B	W	W	W	P	W	W	W	3
W	W	W	W	W	W	W	B	O	B	B	W	W	W	W	P	W	4
W	W	W	W	W	W	W	O	O	B	W	W	W	P	W	P	W	5
W	..	B	B	B	B	A	B	B	..	W	W	..	B	6
O	O	O	E	O	O	B	B	O	O	B	O	O	O	7
O	W	O	O	O	B	B	S	O	O	8
..	..	B	S	S	S	S	A	W	B	A	W	W	W	..	W	W	9
B	B	B	B	B	B	10
W	W	..	A	W	W	W	W	W	B	W	..	W	11
W	W	W	W	W	W	W	O	S	S	W	W	..	W	W	12
W	W	W	B	B	S	A	B	S	..	W	W	..	B	W	S	S	13
W	W	W	W	W	W	W	W	W	P	W	..	W	P	W	P	W	14
W	W	W	W	W	W	W	W	W	W	B	W	W	W	W	W	W	15
B	B	B	B	B	B	B	B	B	B	B	B	B	B	..	S	B	16
B	B	B	B	B	B	B	B	B	B	..	17
B	B	B	B	B	B	..	B	B	B	S	..	B	B	..	B	B	18
..	W	W	B	B	W	B	B	B	B	W	B	..	B	..	B	B	19
W	W	W	W	W	W	W	W	W	W	B	W	W	W	W	20
..	P	B	W	B	B	S	B	B	B	B	S	..	B	W	21
B	B	B	B	B	B	B	B	B	B	B	B	W	B	..	B	W	22
B	B	B	B	B	B	S	B	B	B	..	B	B	23
B	..	B	B	B	B	B	B	B	B	B	B	24

Modified from *Kedzie*.



CHEMICAL APPARATUS.

[See page 74 for description.]

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